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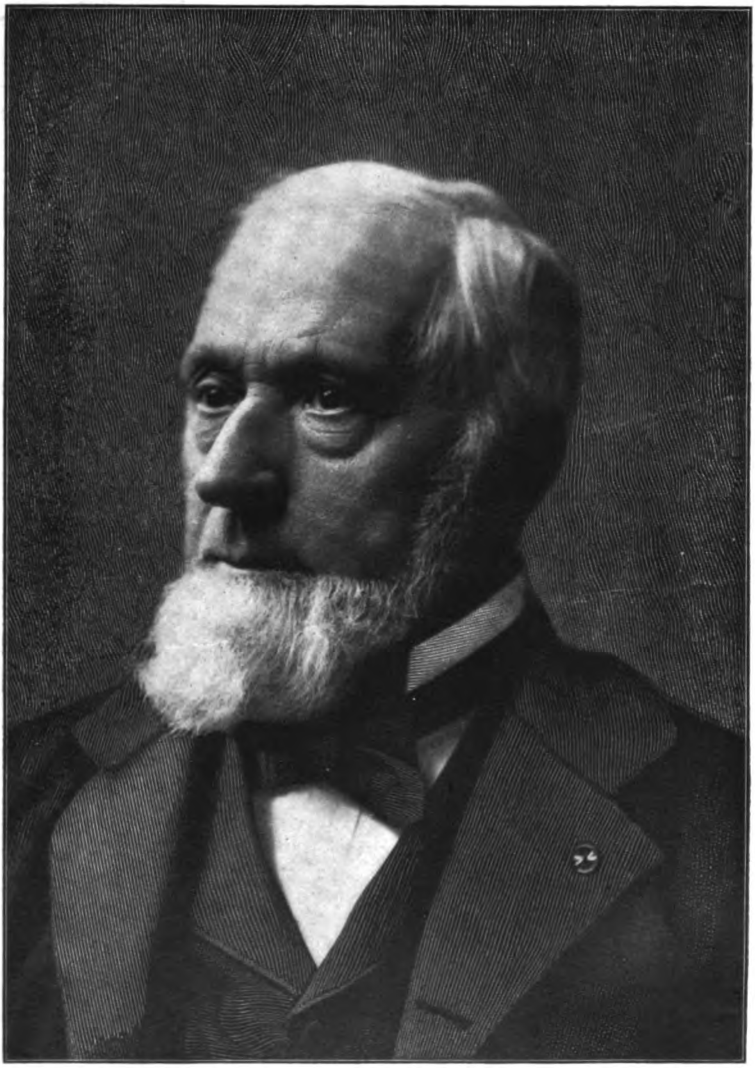

R. C. Kedzie

U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 73.

H. W. WILEY, Chief of Bureau.

PROCEEDINGS

OF THE

NINETEENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,

HELD AT

WASHINGTON, D. C., OCTOBER 2, 3, AND 4, 1902.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1903.

LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., December 15, 1902.

Sir: I have the honor to transmit herewith the manuscript of the proceedings of the nineteenth annual meeting of the Association of Official Agricultural Chemists, with the request that it be published as Bulletin No. 73 of this Bureau.

H. W. Wiley,
Chief of the Bureau of Chemistry and Secretary of the Association of Official Agricultural Chemists.

Hon. James Wilson,
Secretary of Agriculture.

138278

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PROCEEDINGS OF THE NINETEENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

THURSDAY—MORNING SESSION.

The nineteenth annual convention of the Association of Official Agricultural Chemists was called to order by President H. J. Wheeler at 10 o'clock on the morning of October 2, 1902, in the lecture hall of the Columbian University. The following members and visitors were in attendance during the sessions of the convention:

MEMBERS AND VISITORS PRESENT.

Allen, Edwin W., U. S. Department of Agriculture, Washington, D. C.
Angelo, Oliver Perry, U. S. Department of Agriculture, Washington, D. C.

Bache, Arthur Wilbur, U. S. Department of Agriculture, Washington, D. C.
Barnard, Harry E., Chemist State Board of Health, Concord, N. H.
Beal, W. H., U. S. Department of Agriculture, Washington, D. C.
Bigelow, W. D., U. S. Department of Agriculture, Washington, D. C.
Bisbee, Derward B., City Chemist, 7340 Bond avenue, Chicago, Ill.
Blouin, Robert E., Agricultural Experiment Station, Audubon Park, New Orleans, La.
Bosworth, Alfred W., Agricultural Experiment Station, Kingston, R. I.
Breazeale, James F., U. S. Department of Agriculture, Washington, D. C.
Brooke, John C., U. S. Department of Agriculture, Washington, D. C.
Browne, Charles A., jr., Sugar Experiment Station, Audubon Park, New Orleans, La.
Burd, John S., U. S. Department of Agriculture, Washington, D. C.

Chace, E. M., U. S. Department of Agriculture, Washington, D. C.
Carpenter, Frank B., Virginia-Carolina Chemical Company, Richmond, Va.
Carroll, John S., Agricultural College, Miss.
Cavanaugh, George W., Cornell University Agricultural Experiment Station, Ithaca, N. Y.
Chamberlain, Joseph S., U. S. Department of Agriculture, Washington, D. C.
Craighill, George P., John H. Heald & Co., Lynchburg, Va.
Crampton, C. A., Treasury Department, Washington, D. C.

Davidson, R. J., Agricultural Experiment Station, Blacksburg, Va.
Donk, Marion G., Assistant State Chemist, Tallahassee, Fla.
Doyle, Aida M., U. S. Department of Agriculture, Washington, D. C.
Dubois, Wilbur L., U. S. Department of Agriculture, Washington, D. C.

Eaton, Edward N., State Analyst, 1628 Manhattan Building, Chicago, Ill.
Ewell, E. E., U. S. Department of Agriculture, Washington, D. C.

Fraps, George S., Agricultural Experiment Station, Raleigh, N. C.
Frear, William, Agricultural Experiment Station, State College, Pa.

Gore, Herbert C., U. S. Department of Agriculture, Washington, D. C.
Goss, Ralph M., U. S. Department of Agriculture, Washington, D. C.
Gough, T. R., Maryland Agricultural College, College Park, Md.

Hamilton, John, Secretary of Agriculture of Pennsylvania, State College, Pa.
Howard, Burton J., U. S. Department of Agriculture, Washington, D. C.
Harcourt, Robert, Ontario Agricultural College, Guelph, Ontario, Canada.
Hart, E. B., Agricultural Experiment Station, Geneva, N. Y.
Haskins, Henri D., Massachusetts Agricultural College, Amherst, Mass.
Haywood, John K., U. S. Department of Agriculture, Washington, D. C.
Herff, B. von, Director of German Kali Syndicate, New York, N. Y.
Hite, Bert H., Agricultural Experiment Station, Morgantown, W. Va.
Holmes, Charles W., The Columbian University, Washington, D. C.
Hopkins, Cyril G., Agricultural Experiment Station, Urbana, Ill.
Houghton, Harry W., U. S. Department of Agriculture, Washington, D. C.

Jaffa, Myer E., Agricultural Experiment Station, Berkeley, Cal.
Jenkins, Edward H., Agricultural Experiment Station, New Haven, Conn.
Jones, Charles H., Agricultural Experiment Station, Burlington, Vt.

Knight, Carlisle P., U. S. Department of Agriculture, Washington, D. C.
Krug, William H., U. S. Department of Agriculture, Washington, D. C.

La Bach, James O., Agricultural Experiment Station, Lexington, Ky.
Langworthy, C. F., U. S. Department of Agriculture, Washington, D. C.
Lansdale, Harry N., Assistant State Chemist, College Park, Md.
Lawson, H. W., U. S. Department of Agriculture, Washington, D. C.
Leach, Albert E., State Board of Health, Boston, Mass.

McCandless, John M., State Chemist, Atlanta, Ga.
McCarthy, Eugene R., U. S. Department of Agriculture, Washington, D. C.
McDonnell, Curtis C., Agricultural Experiment Station, Clemson College, S. C.
McDonnell, Henry B., Agricultural Experiment Station, College Park, Md.
Magruder, Egbert W., Department of Agriculture, Richmond, Va.
Meng, James S., German Kali Works, 93 Nassau street, New York, N. Y.
Mooers, Charles A., Agricultural Experiment Station, Knoxville, Tenn.
Moore, Charles C., U. S. Department of Agriculture, Washington, D. C.
Morrison, William G., F. S. Royster Guano Company, Norfolk, Va.
Morse, Fred W., Agricultural Experiment Station, Durham, N. H.
Munroe, Charles E., Columbian University, Washington, D. C.
Munson, Lewis S., U. S. Department of Agriculture, Washington, D. C.

Needham, Charles W., The Columbian University, Washington, D. C.
Norton, John H., U. S. Department of Agriculture, Washington, D. C.

Oshima, Kintaro, Middletown, Conn.

Page, Logan Waller, U. S. Department of Agriculture, Washington, D. C.
Patrick, George E., U. S. Department of Agriculture, Washington, D. C.
Patterson, Henry J., Agricultural Experiment Station, College Park, Md.
Penny, C. L., Agricultural Experiment Station, Newark, Del.
Price, Thomas M., Agricultural Experiment Station, College Park, Md.

Reed, Herbert C., Stamford Manufacturing Company, Stamford, Conn.
Robb, John B., Agricultural Experiment Station, College Park, Md.

Ross, B. B., State Chemist, Auburn, Ala.
Runyan, E. G., Inspector of Gas and Meters, Washington, D. C.

Sawyer, Harris E., Consulting Chemist, Boston, Mass.
Seidell, Atherton, U. S. Department of Agriculture, Washington, D. C.
Smith, Bernard H., U. S. Department of Agriculture, Washington, D. C.
Smith, Joseph G., U. S. Department of Agriculture, Washington, D. C.
Spencer, Guilford L., U. S. Department of Agriculture, Washington, D. C.
Stewart Andrew, U. S. Department of Agriculture, Washington, D. C.
Street, John P., Agricultural Experiment Station, New Brunswick, N. J.
Stuart, Duncan, U. S. Department of Agriculture, Washington, D. C.

Teas, William H., Ridgway, Pa.
Terne, Bruno, Union Abattoir Company, Baltimore, Md.
Tolman, L. M., U. S. Department of Agriculture, Washington, D. C.
Trescot, T. C., U. S. Department of Agriculture, Washington, D. C.

Van Slyke, Lucius L., Agricultural Experiment Station, Geneva, N. Y.
Veitch, H. P., U. S. Department of Agriculture, Washington, D. C.

Warner, Harry J., U. S. Department of Agriculture, Washington, D. C.
Watts, Henry C., 850 Drexel Building, Philadelphia, Pa.
Wheeler, H. J., Agricultural Experiment Station, Kingston, R. I.
Wiley, H. W., U. S. Department of Agriculture, Washington, D. C.
Williams, Charles B., Department of Agriculture, Raleigh, N. C.
Wilson, James, Secretary of Agriculture, Washington, D. C.
Wilson, Nathaniel E., Agricultural Experiment Station, Reno, Nev.
Wilson, Herman T., Buena Vista Extract Company, Buena Vista, Va.
Winton, A. L., Agricultural Experiment Station, New Haven, Conn.
Woods, C. D., Agricultural Experiment Station, Orono, Me.

The following order of business was followed:

ORDER OF BUSINESS.

The president's address.

Reports of the referees in the following order:

1. Report on nitrogen.
2. Report on potash.
3. Report on phosphoric acid.
4. Report on soils.
5. Report on ash.
6. Report on foods and feeding stuffs.
7. Report on food adulteration.
8. Report on dairy products.
9. Report on sugar.
10. Report on tannin.
11. Report on insecticides.
12. Reports of special committees (food standards, fertilizer legislation).

The president addressed the convention as follows:

PRESIDENT'S ADDRESS.

GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: An unwritten law seems to have come into existence to the effect that the president of this association is expected to deliver a more or less formal address upon the opening of the convention.

Owing to the fact that new and somewhat arduous executive duties have been added, temporarily, to those which fall to my regular lot, it was but natural that I should seek some precedent for the omission of such an address. It was, therefore, with a feeling of intense satisfaction that I discovered in reading the early records of this association that presidential addresses were not at first in vogue. In fact every president of the association who has been troubled to find the time to prepare a formal address, or who has felt that the grain has already been thrashed from the association "straw," has good reason to place the burden of his complaint upon the shoulders of our present secretary, Mr. Wiley, who, so far as the records show, seems to have been the first president to "address" the association. This address was delivered upon the occasion of the opening of the third convention. Upon the opening of the fifth convention President Chazal omitted the address, since, as he said, "there was a desire to proceed at once to business with the view of accomplishing the objects of the convention in as short a time as possible." Upon the opening of the sixth convention such an excellent address was delivered by our late esteemed friend, John A. Myers, that the custom became firmly established. In lieu of a formal address I shall merely call attention to a few matters which it may be well for us to consider at this time.

Upon reading the proceedings of the eighteenth convention, attention is attracted to several important changes in the methods of business previously in vogue. As a necessary result of the expansion of our work the methods of the "town meeting" have gradually given place to those of a parliamentary body.

Perhaps the most important improvement is the appointment of several committees on "recommendations of referees" instead of a single committee. The results of this change are so gratifying and the necessity of it so obvious to all, that there seems to be little chance that the system will be changed in the near future except by adding new committees as the volume and the character of the work may demand. According to the vote passed at the last convention this committee can only be called upon at this session to report upon minor matters. Important recommendations by referees, such as the adoption of new methods or the radical change of old ones, may be considered by the committees at their leisure, instead of under pressure as heretofore, and the final report is to be rendered, in printed form, if possible, to each member of the association as soon as convenient after the close of the present session. Final action upon the report of these committees can then be taken at the next succeeding convention, or after ample time has been given to all to fully consider and test the proposed changes.

Several important modifications of the constitution, which were made in 1901, were necessitated by the great expansion of the work undertaken, and may be looked upon as the milestones marking the association's path of progress.

It is doubtful if any single point is of greater moment than for us to keep continually in mind that we exist for the purpose of improving and perfecting our methods of analysis, and not for the purpose of testing the work of individual chemists. The older members of the association can readily recall times when this point was seemingly forgotten by referees, to the great detriment of our work.

It can not be too often reiterated that several States in the Union have incorporated in their laws a provision that the results secured by the official methods of this association shall be accepted as final evidence in the courts of the respective States. For this reason as well as from motives of pride in our association we should not be influenced in the adoption and modification of methods solely by the desire to secure an official sanction to some scheme of analysis which will enable us to do our work more quickly or easily, but should exercise due care and act conservatively.

Owing to the complex character of certain investigations undertaken by the association, and the fact that a considerable amount of original research is sometimes demanded, the appointment of a committee to deal with certain subjects rather than a referee and an associate, would seem to be worthy of consideration. It is not infrequently the case that the member of the association who would naturally be selected as referee or associate referee by virtue of his special experience, is too fully occupied to give the matter the attention which is demanded of a referee under the present system. The advice of such a member might, however, be invaluable in formulating lines of investigation and his service might be secured upon such a committee where he might act solely in an advisory capacity.

In connection with the study of methods great attention should be paid in many instances to fundamental principles which tend to affect the final results. As instances of this one might cite the necessity of a careful study of the conditions and circumstances affecting the composition of the "yellow precipitate" obtained in connection with the volumetric determination of phosphoric acid, also the desirability of perfecting methods for the more satisfactory differentiation of the phosphoric acid of soils.

Since the last convention, Congress has officially recognized this association by making it adviser to the Secretary of Agriculture in all matters relating to food standards.

As a result of several inquiries regarding Battle and Dancy's chemical conversion tables, it has been ascertained that the tables are now out of print and that the authors have no intention of issuing a new edition. They have expressed their willingness to have this association make such use of the tables as it desires provided due credit is given to the authors. As a matter of convenience to our members it is recommended that the association consider the advisability of publishing the same or a similar and more comprehensive set of tables for general laboratory use.

Let us in beginning our labors for another year not waste our energies in congratulating ourselves upon the work of the past, but rather look forward to still further energetic and harmonious action, such as has ever tended to enhance the value of our work in the past.

APPOINTMENT OF COMMITTEES.

The PRESIDENT. If I mistake not, the announcement of committees is now in order. It gives me pleasure to appoint the following committee to wait upon the Secretary and Assistant Secretary of Agriculture for the purpose of inviting these gentlemen to attend our meeting:

Messrs. Jaffa, Davidson, and Jenkins.

Committee on resolutions: Messrs. Van Slyke, Frear,[a] and Ross.

Committee on nominations: Messrs. Bigelow, Street, and Jones.

In regard to the committee on nominations, I would like to say that it seems desirable that the election of officers take place, if possible, to-morrow forenoon in order that the executive committee may have time to appoint the referees for next year and announce them before the close of the convention. I would therefore like to suggest that the committee on nominations be asked to report, if possible, to-day, and that we make the election a special order for to-morrow forenoon in order that this may be accomplished.

Committee on amendments to the constitution: Messrs. Frear,[b] Hite, and Hopkins.

I would like to ask if it is the pleasure of the association that the same number of members be appointed on the committees on recommendations of referees as last year. The association will remember that prior to last year we had but one committee on recommendations of referees. Last year a special committee was appointed to propose a subdivision of the subjects. This committee subdivided the work under Committees A, B, and C, Committee A having in charge phosphoric acid, potash, nitrogen, soils, ash, and insecticides; Committee B, dairy products, foods and feeding stuffs, and tannin;[a] and Committee C, food adulteration. Is it the desire of the association that the committees be modified this year, or that they shall remain the same? I should be glad to hear some expression of opinion on this question and await your pleasure as to a motion.

---

[a] Mr. H. B. McDonnell was later appointed in the place of Mr. Frear.

[b] Mr. Van Slyke was afterwards appointed in the place of Mr. Frear.

Mr. Davidson. I move that the same committees be appointed. I think they helped very much in the dispatch of business.

The motion was carried, and the president appointed the following: Messrs. Street, Jaffa, Veitch, Ross, and Runyan, on Committee A; Messrs. Krug, C. A. Browne, Haskins, Munson, and C. H. Jones, on Committeee B, and Messrs. Leach, Winton, Eaton, Barnard, and Tolman on Committee C.

Mr. Wiley. Mr. President, I would like to move that the committee on invitations include the names of the president of this university, Dr. Needham, who was appointed a few months ago, and the dean of the Graduate School, Dr. Munroe. Through their courtesy we are occupying this building, and I think they would appreciate the compliment of an invitation to attend some of our sessions.

The motion was carried, and the committee was instructed to act accordingly.

The President. According to our order of business, the first report to be given is that on nitrogen. Word has been received that the referee on nitrogen will not be here until 10.30 a. m. If it is agreeable to the association we will pass on to the report on potash.

Mr. Wiley. Mr. President, before proceeding with the regular order of business, I would like to say in regard to the committee on food standards that Mr. Frear, the chairman of that committee, is compelled to go away to-night, therefore I move that the report of this committee be received this afternoon.

The motion prevailed.

On motion of Mr. Wiley, the election of officers was set for 11 o'clock Wednesday morning.

Mr. Wiley. I would like to say that the Cosmos Club has extended the courtesy of the club to the members of this association during their stay in the city. The club is on the corner of H and Sixteenth streets, and you will be welcomed there. On entering please register your name in the book, so that we may have a record of those who attend.

The President. It is also with regret that I am obliged to announce that the report of the referee on potash is not ready. It can be presented this afternoon. The next in order will be the report on phosphoric acid.

---

[a] Sugar should have been included in the work of this committee.

REPORT ON PHOSPHORIC ACID.

By C. H. JONES, *Referee.*

Your referee on phosphoric acid has been unable to give the subject delegated to his keeping the attention he desired owing to pressure of work connected with fertilizer and feeding-stuff inspections coming at a time when the preliminary work of formulating trial methods for this year's work should have been done.

Fortunately, however, the total phosphoric acid work has received such careful attention in the past by this association that the methods are in a satisfactory shape and need no overhauling at a referee's hands. The method for the determination of available phosphoric acid is not in as good favor. While ammonium citrate serves fairly well with acid goods it affords low results of doubtful validity with basic slag. Some demand has therefore arisen for a change in the present method of determining the available phosphoric acid content of basic slags. This demand doubtless led your last referee to suggest that further study along these lines was in order.

Contemplated changes from the official method fall naturally into two classes: (1) The substitution for ammonium citrate of another solvent that will be of more general application; (2) the adoption of special solvents for special phosphatic materials. The pros and cons of this question have all been brought to your attention during past sessions and do not merit further attention here.

At the 1900 meeting of this association Mr. Thomas Macfarlane submitted a scheme of citric-acid treatment applicable to commercial fertilizers and a modification of the same fitted for use with basic slag, ground bone, etc. This method is outlined in Bulletin No. 62, Bureau of Chemistry, U. S. Department of Agriculture (p. 50), as well as in Bulletin No. 75 of the inland revenue department, Ottawa, Canada. Mr. Macfarlane shows that different basic slags carry varying amounts of free lime, and that if this be removed before treatment by any method for availability an increased solubility of the phosphoric acid apparently results. He suggests the use of ammonium chlorid solution to remove this lime, and further shows that such treatment does not dissolve the phosphoric acid compounds. These methods are now in use in Canadian fertilizer inspection laboratories, and the results obtained by their use are shown in Bulletins Nos. 75 and 81 of the inland revenue department, Ottawa, Canada. As a rule the so-called insoluble phosphoric acid content is decreased, and consequently that of the citric soluble and available phosphoric acids is increased, particularly in bone fertilizers and slags.

The referee for 1901 was instructed to investigate along these lines. His report contains the results obtained by a citric-acid process, details of which can be seen in Bulletin No. 67, Bureau of Chemistry, U. S. Department of Agriculture (p. 23). The removal of lime by ammonium chlorid solution was, however, not mentioned.

The report of the referee for 1897 also contains the results of comparisons of different methods for determining the availability of basic slag, including modifications of the present ammonium citrate method, Wagner's method, and a 1 per cent citric-acid method.

During the time at his disposal your referee has obtained a few results by different methods, which are here presented:

PHOSPHORIC ACID IN BASIC SLAG.

		Per cent.
1.	Total phosphoric acid found	17.56
	Available phosphoric acid found by—	
2.	Direct treatment "official" ammonium citrate method	5.22
3.	Substituting 100 cc 1 per cent citric acid for ammonium citrate	4.72
4.	Substituting 100 cc 2 per cent citric acid for ammonium citrate	7.21
5.	Substituting 100 cc ammonium citrate+100 cc water	4.54

				Per cent.
6.	Washing 2 grams on filter with 200 cc water.		Residue treated as per (2)	6.94
7.			Residue treated as per (5)	7.00
8.			Residue treated as per (3)	4.98
9.			Residue treated as per (4)	8.17
10.	Washing 2 grams on filter with 200 cc 5 per cent sugar solution.		Residue treated as per (2)	6.44
11.			Residue treated as per (5)	6.80
12.			Residue treated as per (3)	5.74
13.			Residue treated as per (4)	8.93
14.	Washing 2 grams on filter with 200 cc 5 per cent ammonium chlorid solution.		Residue treated as per (2)	6.89
15.			Residue treated as per (5)	7.36
16.			Residue treated as per (3)	6.63
17.			Residue treated as per (4)	9.06
18.	Boiled 2 grams with 100 cc 5 per cent ammonium chlorid solution, replacing evaporated water.		Residue treated as per (2)	8.70
19.			Residue treated as per (3)	7.40
20.			Residue treated as per (4)	9.14
21.	Rotated 30 minutes with 200 cc.	Water (room temperature),	Residue treated as per (2)	7.22
22.		5 per cent ammonium chlorid.	Residue treated as per (2)	8.04
23.	Macfarlane method.	Boil 2 grams with 100 cc 5 per cent ammonium chlorid solution. Filter. Add to residue 100 cc 1 per cent citric acid. Shake. Add 100 cc more 1 per cent citric acid. Shake every five minutes for half an hour. Filter; wash. Determine phosphoric acid in residue.	Temperature, 20° C	10.44
24.			Temperature, 30° C	10.51
25.			Temperature, 40° C	10.90
26.			Temperature, 50° C	10.75

The results, it is seen, run from 4.54 per cent to 10.90 per cent, depending on the method of procedure. With the Macfarlane method the variation in results between 20° and 50° C. is not marked, and indicates that differences in ordinary room temperatures have only a slight influence. The last four results were obtained by difference between the total phosphoric acid present and that remaining in the insoluble residue. The direct percentages obtained by determining the phosphoric acid in the filtrate were 10.36, 10.83, 11.06, and 10.88, respectively. They show fair agreement, and a direct estimation would often save considerable time.

On boiling 2 grams of this slag in 100 cc of 5 per cent ammonium-chlorid solution for thirty minutes and replacing evaporated water, there was removed 11.87 per cent of lime (CaO). Boiling in 200 cc of a similar solution removed 13.35 per cent of lime (CaO). No phosphoric acid was found in the filtrates from the above. Washing 2 grams on a filter with 200 cc of the ammonium chlorid solution removed 6.42 per cent of lime, while a similar washing with 5 per cent sugar solution dissolved only 1.85 per cent of lime. The sample sent out by the referee in 1901 gave an availability of 11.57 per cent by the Macfarlane method.

It is well recognized that the solvent power of the sundry solutions employed in availability determinations depends upon the quantity of the solvent and material used and the length of time allowed for its action. Temperature also plays its part. This being true, it would seem desirable to at least agree on the per cent of the total phosphoric acid present we are willing to concede as available in a sample of basic slag of standard composition and fineness. For example, let us say that the Wagner method shows 80 per cent available phosphoric acid and the present ammonium citrate method 30 per cent. There may be those present who believe the former is too high, while I think we are all agreed that the latter is too low. The happy medium therefore lies between 30 and 80 per cent, and if once agreed upon it would, in my opinion, prove of great help in the selection of a special method for the availability of phosphoric acid in basic slag.

The **President**. Before proceeding to the next subject I would like to announce changes in committees necessitated by Mr. Frear's being called away. I appoint in his place Mr. McDonnell, on the committee on resolutions, and as chairman of the committee on amendments to the constitution, Mr. Van Slyke. Next in order will be the report on ash.

REPORT ON ASH.

By G. S. Fraps, *Referee.*

Before sending out samples or requests for cooperation the referee undertook the following work on the determination of sulphur:

Preliminary Work on Sulphur.

The following methods were studied:

(1) *Nitric-acid method* (*the provisional method of the association*).

Five grams of material are placed in a 3½-inch porcelain evaporating dish, 20 cc of nitric acid (concentrated) added, and the mixture heated cautiously on the water bath until all danger of overflowing has passed. It is then partly evaporated, 10 cc of a 5 per cent solution of potassium nitrate is added, the mixture evaporated to complete dryness, and ignited, at first gently, then under a blast lamp until the residue is white. It is then dissolved in hydrochloric acid, evaporated to dryness, and heated for some time in an air bath to render silica insoluble. The residue is taken up in water with the addition of a little acid, filtered, and the sulphuric acid precipitated with barium chlorid in the usual way.

This method gives good results. It has a disadvantage, however, in that the ash produced is easily fusible, acts upon the porcelain dish, and is ignited free of carbon with great difficulty. It was to overcome this objection that the modification described below was tested.

(2) *Nitric-acid method modified.*

The modification consists in the use of 20 cc of a solution of calcium acetate containing 0.56 gram of calcium oxid (CaO), in place of 20 cc of potassium-nitrate solution. The ignition takes place much more easily, the ash does not fuse, and is more easily burned free of carbon than when potassium nitrate is used.

(3) *Boiling with nitric acid.*

An attempt was made to destroy the organic matter by boiling the material with nitric acid, which I understand is the method used for meats in the nutrition investigations of the Office of Experiment Stations. The method did not succeed with vegetable materials. Several experiments were made, but in no case was all organic matter of the vegetable materials destroyed, even when 5 grams of material was boiled to dryness with 100 cc nitric acid in a Kjeldahl flask. As it would, therefore, be necessary to wash the residue from the flask into a dish, and ignite with the addition of potassium nitrate or calcium acetate to remove organic matter, this method would differ from the two preceding only in that a larger quantity of acid is used and the digestion with the acid is more prolonged. The method was not further examined.

(4) *Fusion with caustic soda.*

This is the standard method for determining sulphur in nonvolatile organic compounds. The method as tested was as follows:

Five grams of material in a nickel crucible were saturated with 40 cc of a solution of 150 grams sodium hydroxid in 300 cc water, evaporated to dryness, and fused with the addition of a little sodium peroxid. The fusion requires care to prevent the material from overflowing the dish, and the whole process is somewhat long. The melt was dissolved in water, neutralized and acidified with hydrochloric acid, evaporated to dryness and heated to render silica insoluble, filtered, and the sulphuric acid precipitated from the filtrate, the volume of which was about 200 cc. The sodium hydroxid and sodium peroxid contained a large quantity of sulphates; about 0.1 gram in the quantity used in the determination was found in the blank determination. The results with this method are decidedly low. This may possibly be due to the solubility of barium sulphate in sodium chlorid, about 38 grams of the latter salt being present in the solution from which the barium sulphate was precipitated, or it may be owing to the error caused by impurities in the reagents.

(5) *Eschka's method.*

This is the method usually employed for the determination of sulphur in coals. It was applied to plant materials as follows:

Five grams material were intimately mixed, in a platinum dish, with 7½ grams of a mixture of two parts magnesium oxid to one of dried sodium carbonate. The mixture was heated, stirring frequently, and the temperature raised slowly until all carbon appeared to be burned off. The mass was transferred to a beaker with about 50 cc water; 15 cc of saturated bromin water was added and boiled five minutes. It was allowed to settle, decanted through a filter, boiled a second and third time with about 30 cc water, and washed thoroughly. The filtrate was made acid with hydrochloric acid, boiled until bromin was expelled, and then precipitated with barium chlorid in the usual way. The results by this method were too low.

RESULTS OF WORK.

The analytical results by the different methods are given in the following table (Table I). The figures in each case are corrected for the sulphur in the reagents found by a blank determination. The blank was very low in the case of the two nitric-acid methods. In the Eschka method it was equivalent to 0.0058 gram barium sulphate in the quantity of the reagents used for each test. As has already been stated, the fusion method gave results much too low, and the blank was high, being 0.1103 gram barium sulphate.

TABLE I.—*Determination of sulphur (SO_3) by different methods.*

	Nitric-acid method.	Nitric-acid method modified.	Eschka method.
	Per cent.	*Per cent.*	*Per cent.*
Corn	0.249	0.287	0.238
	.244	.286	.208
Average	.247	.287	.223
Peas	.481	.480	.297
	.533	....	.312
	.503	....	.313
Average	.518	.480	.307
Millet	.369	.427	.451
	.376	.434	.460
	.400	....	....
Average	.382	.431	.456

OFFICIAL WORK ON SULPHUR.

It was decided to compare the provisional method for the determination of sulphur with the method modified by the use of calcium acetate in place of potassium nitrate. A comparison between two methods for potash was also determined. The following circular letter was sent out to a number of members of this body:

RALEIGH, N. C., *May 1, 1902.*

DEAR SIR: The referee on ash for the present year will continue the study of the methods of determining sulphur and potash in plant materials. It is thought best to work out one portion of the problem at a time. Kindly inform the referee if you will be able to cooperate. The samples will be sent out about the latter part of May. The work of last year proves conclusively that the sulphur in an ash, as ordinarily prepared, is no indication of the amount of sulphur in the plant, which may be vastly greater. Kindly give us the benefit of any suggestions.

Very truly,

G. S. FRAPS, *Referee.*
FRANK T. SHUTT, *Associate Referee.*

Eight chemists requested samples. R. J. Davidson, of Virginia, sent the following table of analyses:

TABLE II.—*Determination of sulphur* (SO_3) *by different methods* (*Davidson*).

Method.	Dried apples, sample 1.	Dried apples, sample 2.	Bran.
	Per cent.	*Per cent.*	*Per cent.*
Ignition with calcium acetate	0.09	0.10	0.27
Nitric-acid method	.09	.10	.45
Eschka's method	.10	.11	
In ash			.09

W. P. Headden, of Fort Collins, Colo., wrote as follows:

> If you will refer to my bulletin on alfalfa, No. 35, of this station, pages 84–87, you will see that I did some work in this line in 1895 in regard to chlorin, sulphur, and phosphorus, but did nothing in regard to loss of potash. I found a very considerable loss of chlorin and sulphur, but none for phosphoric acid. I was rather surprised at the latter, as we know that phosphates heated with carbon, especially if there is sand enough present to set phosphoric acid free, may be reduced. In my experiments there were doubtless enough basic salts present to avoid such reactions; besides, the temperature of incineration was very low—as low as would accomplish the incineration.
>
> You state that the sulphur in the plant may be vastly greater than that indicated by the determination of sulphuric acid or total sulphur in the ash. You will see that I found in one instance 4.88 per cent in the ash and 8.09 per cent in the plant, almost 66 per cent more.

Mr. Charles P. Beistle wrote as follows:

> I would say that I have done considerable work on the determination of sulphur in plant substances and proteid bodies, and have found the only satisfactory way is by fusion in a mixture of sodium hydrate and potassium nitrate. Among other methods used were ignition in a closed vessel containing oxygen under about 20 atmospheres pressure, ignition in a combustion tube in a current of oxygen gas, passing products of combustion through bromin water, and digestion in nitrohydrochloric acid. The first-mentioned method always gave the highest results. The next two methods gave almost as high results, but required special apparatus and a great expenditure of time. To determine sulphur in the ash of plants gives no indication whatever of the amount of sulphur present in the original material, and the calcium-acetate method (simple ignition with calcium acetate), although giving higher results, is of no real value as a method of estimating total sulphur.
>
> I would suggest that fusion with sodium peroxid might be successfully used as a method of oxidizing the plant material and changing sulphur to sulphate.

WORK ON ASH.

Two samples were prepared, corn bran and cotton-seed meal, and sent out with the following letter:

DIRECTIONS FOR WORK.

The samples consist of corn bran and cotton-seed meal. Determine moisture (in the usual way), sulphur, and potash in both samples. Make at least two determinations by each method. In reporting give weights of precipitates, per cent on samples as received, and on dry matter. Please report results as early as possible.

Method I.—Sulphur.

Place 5 grams material in a 2½-inch porcelain evaporating dish, add 20 cc concentrated nitric acid, and heat the mixture cautiously on the water bath until all danger of overflowing has passed. Then evaporate partly, add 10 cc of 5 per cent solution of potassium nitrate, evaporate to complete dryness, and ignite, at first gently, then more vigorously until the residue is white. The residue is then dissolved in hydrochloric acid, evaporated to dryness, and heated for some time in an air bath to render silica insoluble. The residue is taken up in water with the addition of a little hydrochloric acid, filtered to 150 cc or more, and the sulphuric acid precipitated with barium chlorid in the usual way. Make a blank test with the reagents.

Method II.—Sulphur.

The same as the preceding, substituting 20 cc of a solution of calcium acetate for the potassium nitrate. The solution is prepared by dissolving 12.5 grams calcium carbonate, chemically pure, in chemically pure acetic acid, and diluting to 250 cc. Make a blank test with the reagents.

Method III.—Potash.

Determine by ignition with sulphuric acid as in the methods of the A. O. A. C., page 22 b. With the bran use a quantity of the solution corresponding to at least 2 grams of bran.

Method IV.—Potash.

Char 10 grams substance in a platinum dish, extract twice with hot dilute hydrochloric acid 1:10, and wash, receiving the extract and washing in a 500 cc flask. Return filter and contents to the dish, and ignite to whiteness. Dissolve the residue in hydrochloric acid, add to the extract in the flask, make up to volume, and determine potash as in Method III.

Report potash as K_2O, sulphur as SO_3. Please give the referee the benefit of your suggestions in regard to the work.

MOISTURE.

The moisture determinations on the samples sent out are given simply as a matter of record (Table III). The agreement is satisfactory.

TABLE III.—*Moisture determinations.*

Analyst.	Corn bran.	Cotton-seed meal.
	Per cent.	*Per cent.*
J. H. Gibboney, Virginia	12.10	7.28
E. G. Runyan, Washington, D. C.	12.67	7.20
Frank T. Shutt, Ottawa	12.30	6.97
A. T. Charron, Ottawa	12.55	7.08
R. W. Thatcher, Washington	13.02	7.96
	13.07	8.01

DETERMINATION OF SULPHUR.

The results obtained for sulphur in the samples as sent out are given in the following table:

TABLE IV.—*Determination of sulphur* (SO_3).

Analyst.	Corn bran.		Cotton-seed meal.	
	Provisional method.	Modified provisional method.	Provisional method.	Modified provisional method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
J. H. Gibboney, Virginia	0.27	0.32	1.06	1.07
	.28	.34	1.07	1.10
	.28	.30	1.01	1.00
E. G. Runyan, Washington, D. C.	.43	.33	1.04	1.08
	.42	.34	1.05	1.08
	.44	.32	1.04	1.14
Frank T. Shutt, Ottawa	.40	.15	1.27	1.10
	.40	.13	....	1.05
A. T. Charron, Ottawa	.34	.24	1.24	1.00
	.34	.24	1.34	1.08
G. S. Fraps, North Carolina	....	.41	....	1.03
	....	.40	....	1.00
	....	....	....	1.03
	....	....	....	1.08
R. W. Thatcher, Washington	.27	.24	.99	.97
	.23	.25	1.00	.97

REMARKS OF ANALYSTS.

R. J. Davidson, Virginia.—We tried several determinations on these substances by Eschka's method, but found the result too low. On 2-gram samples we got the following weights of barium sulphate: 0.0240, 0.216, and 0.0209 grams, just about one-third of what they should be.

R. W. Thatcher, Washington.—My experience with the two methods suggested for the determination of sulphur is that they give practically identical results, those obtained by the calcium acetate ignition method being very slightly lower. The use of calcium acetate allows much more rapid burning to whiteness, since the potassium nitrate present in the other method fuses very easily, and thus incloses particles of carbonaceous matter, which are finally removed only after a somewhat tedious process.

Frank T. Shutt, Central Experimental Farm, Ottawa, Canada.—We found great difficulty in obtaining a white ash by Method II. Even after prolonged ignition the ash remained gray, and on treatment with acid showed the presence of carbon. You will notice that invariably higher results were obtained by Method I. As far as one can judge from duplicate determinations and general conduct and appearance of the material during treatment, Method I seems to be very satisfactory. To lessen the danger from loss while frothing (treatment with nitric acid on water bath) a 3-inch porcelain evaporating dish was used. It will be found much safer than a 2½-inch dish. Ignition in a muffle furnace proved easier and quicker than with the blast lamp.

The agreement between the figures obtained by different analysts by both methods is not satisfactory. The trouble is with the methods. They are not very accurate. As will be shown below, the results given by the nitric acid method are much lower than they should be.

WORK OF H. C. SHERMAN ON SULPHUR AND PHOSPHORUS.

H. C. Sherman, of Columbia University, New York City, during the past summer has been studying the methods for the determination of sulphur and phosphorus in organic materials. We have had some correspondence on the subject, and he has kindly communicated to me the results of his work, which will appear in the Journal of the American Chemical Society. The methods which Dr. Sherman has studied are as follows:

(1) Combustion in oxygen under pressure of 25 to 30 atmospheres in the bomb calorimeter.

(2) Fusion with alkali, as described by Osborne.

(3) The provisional method of this association (the nitric acid method), with the use of 2 grams of sample.

Six samples of material were used. In every case the nitric acid method gave lower results than the other two methods, the average amount of sulphur found being about five-sixths of that present. It seems probable that the greater part of the discrepancy is due to volatilization of sulphur in some form of combination not sufficiently oxidized to stand the heat generated at this point. Such a loss could doubtless be avoided by repeated evaporation with nitric acid, or by the addition of alkali along with the nitrate, but the method would thus become practically that of Salkowski. Combustion in oxygen has the advantage over the fusion method.

The fusion method is described by Osborne as follows:[a]

> About 10 grams of sodium peroxid were converted into hydroxid in a nickel crucible by adding a little water and boiling over an alcohol lamp until the excess of water was expelled. From 1 to 2 grams of the protein were then stirred into the slightly cooled hydroxid and oxidized by gradually raising the heat and adding small portions of sodium peroxid until the oxidation was complete. The fused mass was then dissolved in 400 cc of water, its solution strongly acidified with hydrochloric acid, boiled until the excess of peroxid was destroyed and chlorin expelled, filtered, made neutral with ammonia, and an excess of 4 cc of concentrated hydrochloric acid added. From the boiling solution sulphuric acid was precipitated by gradually adding a solution containing 1 gram of barium chlorid. After standing overnight on a steam table the barium sulphate was filtered out, washed, ignited, and weighed.

Dr. Sherman uses 15 grams of sodium peroxid, and with vegetable materials the acidulated solution was evaporated to dryness, and heated, to dehydrate any silica which might have been present.

This method differs from that tested by the referee chiefly in the use of a larger volume of water in the solution from which the barium sulphate was precipitated. As it is well known that barium sulphate is soluble in strong solutions of salts, it is possible that this may account for the difference.

Dr. Sherman exchanged samples with the referee, with the following results. The sample of lean meat was sent by Dr. Sherman. The other two samples are the referee's samples.

TABLE V.—*Comparison of methods for the determination of sulphur.*

Method and analyst.	Lean meat.	Cotton-seed meal.	Corn bran.
	Per cent S.	*Per cent SO_3.*	*Per cent SO_3.*
Compressed oxygen method—Sherman	0.82	1.49	0.47
Fusion method—Sherman	.81	....	....
Nitric acid method—Sherman	.68	....	....
Nitric acid method—Fraps	.57	1.03	.40
	.52	1.07	.41

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[a] Jour. Am. Chem. Soc. 24: 142 (1902).

I am convinced by this work that the nitric acid method gives too low results.

DETERMINATION OF POTASH.

The results obtained by the different analysts for potash by the methods already described are as follows:

TABLE VI.—*Potash determinations.*

Analyst.	Corn bran.		Cotton-seed meal.	
	Method I—Sulphuric acid.	Method II—Charring and extracting.	Method I—Sulphuric acid.	Method II—Charring and extracting.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
J. H. Gibboney	0.43	0.39	1.80	1.82
	.42	.39	1.78	1.82
E. G. Runyan	.37	.38	1.61	1.73
	.38	.36	1.56	1.68
	....	....	1.60	1.72
	....	....	1.64	1.77
R. W. Thatcher	.42	.37	1.74	1.64
	.45	.38	....	1.72

There seems to be a slightly larger quantity of potash obtained by charring and extracting than by ignition with sulphuric acid, though the results are not conclusive.

DETERMINATION OF SULPHATES.

It has been suggested to the referee that not the total sulphur but the sulphur existing as sulphates in plants should be considered as the sulphur of the ash, since the organic sulphur would otherwise be counted twice. It is necessary, however, that we should have a method for the determination of total sulphur, in order to estimate the quantity of that element required by the growing plant; the fact should not be lost sight of for an instant that the determination of sulphur in an ash, as heretofore made, throws no light whatever upon the quantity of sulphur in the plant, and, since it leads to false conclusions, is worse than valueless.

That part of the sulphur in plants exists as sulphates, and part in the organic form, has been known for a long time. R. Arendt, in 1856, was the first to distinguish between organic and inorganic sulphur in plants and to estimate total sulphur with any degree of accuracy. He extracted the sulphates with acidulated water, and estimated the total sulphur by fusion with alkalies. Ulricht pursued a similar plan. Arendt (1857) found small amounts (0.04–0.06 per cent) of sulphates in the lower leaves and ears of the oats, and more in the upper leaves (0.22–0.44 per cent). Ulricht (1859) states that sulphates are totally absent from the lower leaves and stems of red clover, while present in the upper leaves and the blossom.

Wolff and his associates (1859) give the following quantity of sulphates and organic sulphur as present in the dry matter of green rape.

TABLE VII.—*Distribution of sulphur in green rape (Wolff).*

Part of plant.	Organic sulphur.	Sulphates.
	Per cent S.	*Per cent SO*$_3$.
Seeds	1.127	0.020
Straw	.167	.021
Chaff	.310	1.464

Wolff and Yelin (1860) did not find any sulphates in the grain or straw of wheat, in barley, oats, or lucerne. Knop and Ritter (1859) found sulphates in garden beans (0.14 per cent) but none in the bean straw.

E. Schulze[a] investigated the changes in sulphur during the sprouting of the seeds of the yellow lupine. The finely ground material was completely extracted with warm water, acidified with hydrochloric acid after separating a small quantity of albumen, and precipitated with barium chlorid. As the precipitate did not appear pure it was fused with sodium carbonate with the addition of a little potassium nitrate, the melt extracted with water, and the sulphuric acid precipitated with barium chlorid. The seed contained 1.028 per cent sulphur, 0.154 per cent as sulphates (0.385 per cent calculated as SO_3). The sprouted seeds contained 1.510 per cent and 1.703 per cent sulphur as sulphates after 12 and 15 days, respectively. An oxidation of organic sulphur took place during the germination.

Tammann[b] obtained 0.359 per cent SO_3 from peas, by fusion with sodium carbonate and saltpeter, and 0.075 per cent SO_3 precipitated by barium hydroxid from the hot water extract purified with tannic acid. After germinating 25 days, 0.191 per cent SO_3 was present—a decided increase. By boiling the alkaline filtrate from the barium sulphate with hydrochloric acid a second precipitate appeared, due, he says, to the presence of ethereal salts of sulphuric acid. The quantity of this second precipitate from the ungerminated seeds was insignificant as well as from the seeds germinated in darkness, while from those germinated in the light it corresponded to 0.019 per cent SO_3.

Berthelot and Andre[c] investigated the sulphur in different forms in plants at different stages of growth. They concluded that the sulphur content of the plant increases continuously until it blossoms. The amount of sulphur present in organic compounds reaches its maximum at the time of flowering, and then gradually decreases partly on account of reoxidation of the sulphur, partly by elimination of volatile sulphur compounds. The proportion of the two forms of sulphur in the seeds varies considerably in different species of plants. In *Avena sativa* nearly all the sulphur is present in the organic form; on the other hand, in the white lupine only 6.7 per cent of the total sulphur is in organic combination.

I have gone thus fully into the literature in order to give a clear idea as to the present standing of the determination of sulphates in plants. It is seen that the quantity of sulphates present may vary considerably, both in the seeds of different plants, different parts of different plants, different parts of the same plants, and in the same plant at different stages of growth. At times, indeed, the amount of sulphur present as sulphates is very small.

The following method was used for the determination of sulphates in several materials:

> Five grams substance was mixed well with 50 cc of a 1 per cent solution of hydrochloric acid, allowed to stand half an hour, filtered, and washed with the dilute acid to 250 cc or more. The liquid was heated to boiling, barium chlorid was added, and the determination completed in the usual way.

Instead of the 1 per cent hydrochloric acid, water was used in several cases, but it was found to dissolve organic matter which was afterwards precipitated on coming in contact with the acid.

No sulphur as sulphates was found in the following materials: Corn, peas, green millet, timothy hay, corn silage, peanuts.

Sulphur as sulphates was found in the following: Oats (trace), crimson clover straw, 0.003 per cent; cotton-seed meal, 0.001 per cent; green cowpea vines, 0.085 per cent.

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[a] Landw. Versuchs. Stat. **19:** 172 (1876).

[b] Ber. d. chem. Ges. **19:** 261 (1886) Ref.

[c] Biedermann's Central-Blatt, 555 (1891).

DETERMINATION OF CHLORIN.

The writer has already called attention to the fact that chlorin as well as sulphur will be lost in burning a plant substance to an ash by itself. It will appear later that the loss is due to decomposition of the chlorids by the heated organic matter.

Chlorin was determined in a number of substances in two ways, namely, in the ash as prepared in the usual way and by fusion of the plant material with sodium hydroxid, a method which will be described in full later on in this paper. The results of the determination are as follows:

TABLE VIII.—*Chlorin in plants.*

Material.	Ash method.	Sodium hydroxid method.
	Per cent.	*Per cent.*
Corn	Trace.	0.040
Peas	.005	.008
Peas, No. 2	.001	.012
Oats	.005	.097
Oats, No. 2	.001	.056
Peanuts	.008	.017
Cotton-seed meal	.008	.032
Cotton-seed hulls	.005	.013
Linseed meal	.010	.029
Millet	.120	.187
Corn silage (dry)	.310	.607
Green rape (dry)	1.261	1.290
Green peas (dry)	.160	.205
Sweet potatoes (dry)	.341	.362
Timothy hay	.864	.888

It is evident that a considerable proportion of the chlorin in plants may be lost in burning them to an ash, particularly when there is only a small quantity of chlorin in the plant.

The cause of the loss is shown by the work of Davies, reported in the Journal of the Society of Chemical Industry.[a] He experienced similar losses when sugar, starch, or filter paper was ignited with sodium chlorid. The alkalinity of the ash was evidence that no base had been lost, and if the sodium chlorid was added after the sugar had been charred, and the ignition then completed, no loss took place; that is to say, the loss took place during the initial stages of the combustion, and not during the time the carbon was being burned off, and was probably due to the decomposition of the chlorids by the hot vapors from the organic matters. Extracting the material with hot water after charring and then completing the combustion can, therefore, have no effect on the loss of chlorin, since it takes place before the extraction. When sodium carbonate equivalent to 5 per cent of the organic matter was added to the material, the loss of chlorin was prevented.

Three methods for the determination of chlorin in plants were tested.

(1) *Ignition of the material with calcium acetate.*

Five grams substance were moistened with 20 cc of a solution of calcium acetate containing 0.56 gram calcium oxid, evaporated to dryness, and ignited. The ash was dissolved in nitric acid, filtered, and chlorin determined in the filtrate by the Volhard method. On account of the very small quantity of chlorin present in many of the substances, a gravimetric method could not be used as the precipitate would not coagulate. The results by this method are low (Table IX).

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[a] Jour. Soc. Chem. Ind., **20**: 98 (1901).

(2) *Fusion with sodium hydroxid.*

Five grams substance were mixed thoroughly with 10 cc of a solution of 100 grams caustic soda in 200 cc water, evaporated to dryness, and finally fused. A nickel crucible was used for a portion of the work and silver crucibles for the remainder, due care being taken to prevent silver from being detached from the crucible. It is better to use nickel crucibles, as there is always a little danger of small particles of silver being detached. The melt was dissolved in water, neutralized with nitric acid, filtered, and chlorin determined in the filtrate by the Volhard method.

The method yields satisfactory results, but it is long, and the crucibles wear out rapidly.

(3) *Ignition with sodium carbonate.*

Five grams substance in a platinum dish are impregnated with 20 cc of a 5 per cent solution of sodium carbonate, evaporated to dryness, and ignited as thoroughly as possible. The residue is extracted with hot water, filtered, and washed. It is returned to the platinum dish, ignited to an ash, dissolved in nitric acid, and the chlorin determined by the Volhard method.

In one or two cases the filtrate from the first extraction was yellow from organic matter. It was returned to the dish with the charred residue, evaporated to dryness, and ignited. The residue burned very readily to a white ash. The sodium carbonate method gives as accurate results as the method of fusion with caustic soda, and as it is shorter and easier, it is to be recommended for use.

The results by the three methods are contained in the following table:

TABLE IX.—*Methods for chlorin.*

Material.	Ignition with calcium acetate.	Fusion with caustic soda.	Ignition with sodium carbonate.
	Per cent.	*Per cent.*	*Per cent.*
Corn	0.002	0.040	0.027
	....	.040	.026
Millet	.148	.165	.188
	....	.171	.194
	....	.204	....
Corn silage	.400	.616	.610
	....	.599	....
Cotton-seed meal	.031	.032	.020
Cotton-seed hulls	.010	.013	.017
Green peas	.215	.197	.228
	....	.212	.239
Oats	.034	.056	....
Timothy hay	.870	.914	.897
	....	.878	.911
	....	.872	....

RECOMMENDATIONS.

The referee makes the following recommendations:

(1) Either that the method of fusion with sodium hydroxid and sodium peroxid be adopted as a provisional method by this association, or else that the referee on ash be requested to make a further study of the fusion method.

(2) That the method of determining chlorin by ignition with sodium carbonate be adopted as a provisional method.

The PRESIDENT. Next in order will be the report on potash. We will now listen to that report.

REPORT ON POTASH.

By H. B. McDONNELL, *Referee.*

The work on potash was not taken up until after the appearance of the proceedings of the association for last year, and the work was necessarily planned rather hurriedly and without the careful preliminary work that is desirable.

Early in August a circular letter was sent out inviting cooperation in the work, and favorable replies were received from 12 laboratories, with requests for samples. Reports have been received from 9 laboratories, so that, including that of the referee, 10 laboratories are represented in the work, and 14 chemists.

It was recommended at the meeting last year (1) that the causes of low results obtained by the official method be investigated, (2) that the milk-of-lime method be submitted for investigation for the ensuing year, and (3) that our method of determining moisture in potash salts be compared with the German method.

The following directions were sent out with the samples:

DIRECTIONS FOR THE A. O. A. C. POTASH WORK, 1902.

The bottles sent, with samples to be prepared therefrom, are as follows:

Bottle 1, K_2SO_4, chemically pure; bottle 2, kainit; bottle A, acid phosphate; bottle B, blood; bottle C, fish.

Sample 1, K_2SO_4, chemically pure.
Sample 2, kainit.

$$\text{Sample 3}\begin{cases}\text{10 per cent No. 1.}\\ \text{90 per cent A.}\end{cases}$$

$$\text{Sample 4}\begin{cases}\text{25 per cent No. 2.}\\ \text{75 per cent A.}\end{cases}$$

$$\text{Sample 5}\begin{cases}\text{10 per cent No. 1.}\\ \text{50 per cent A.}\\ \text{20 per cent B.}\\ \text{20 per cent C.}\end{cases}$$

$$\text{Sample 6}\begin{cases}\text{25 per cent No. 2.}\\ \text{40 per cent A.}\\ \text{20 per cent B.}\\ \text{15 per cent C.}\end{cases}$$

Compare flasks with pipette before use.

Ingredients to be weighed separately into graduated flasks. Total charge, 10 grams for 500-cc flasks and 5 grams for 250-cc flasks.

Note if 250-cc or 500-cc flasks are used. Use former size for first test, the latter for duplicate. Referee recommends the optional use of 250-cc flask and 5-gram charge.

Report result of blank test on $PtCl_4$ solution with number of cubic centimeters.
Report cubic centimeters $PtCl_4$ used, and strength.

Test samples 1 and 2 by the official method.

Test samples 3, 4, 5, and 6 by Lindo-Gladding method.

Test samples 3, 4, 5, and 6 by milk-of-lime method, as used last year.

Test samples 3, 4, 5, and 6 by the following method: Boil with water as in the Lindo-Gladding method, add ammonia in slight excess, then $BaCl_2$ solution in slight excess, then ammonium carbonate solution in slight excess to precipitate excess of barium; cool, make up to volume, mix, filter a portion, using a dry filter, transfer 50 cc to a dish (may be of porcelain), evaporate to dryness, heat carefully on hot plate or in air bath (so that loss will not occur by decrepitation on ignition), ignite to dull redness over Bunsen burner, add about 25 cc of hot water, filter and wash into a dish, add excess of platinum solution, evaporate and treat as usual.

Determine moisture in 1 and 2 by official method.

Determine moisture in 1 and 2 by heating to dull redness.

The German method for concentrated salts is to heat 10 grams of the sample in a covered platinum crucible for about 10 minutes, with small flame to dark-red heat. In salts rich in $MgCl_2$ avoid decomposition and loss of chlorin by covering the substance with freshly ignited lime, or else the chlorin is determined before and after ignition, and correction is made for its loss, paying attention at the same time to the absorption of oxygen.

Referee will test A, B, and C for potash, and will determine moisture in 1 and 2, and also in a few samples of commercial potash salts, by heating in a combustion tube and absorbing the water in $CaCl_2$.

Please have results in by September 27, as the association will meet on October 2.

H. B. McDonnell, College Park, Md.,
Referee.
H. D. Haskins, Amherst, Mass.,
Associate Referee.

Results of Cooperative Work on Potash.

The results reported are given in detail in the following tables:

Table I.—*Results of determination of potash in samples.*

Analyst.	Official method.		Lindo-Gladding method with HCl.			
	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Sample No. 5.	Sample No. 6.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
H. B. McDonnell, Maryland	53.98	12.22		3.07		
	54.25	12.12		3.07		
Mean	54.12	12.17				
T. R. Gough, Maryland	54.55	11.96				
	54.55	12.08				
Mean	[a] 54.55	12.02				
H. D. Haskins, Massachusetts	[a] 53.39	11.95				
M. H. Pingree, Pennsylvania	54.00	12.20				
	54.10	12.24				
Mean	54.05	12.22				
John S. Burd, U. S. Dept. Agr	53.94	12.04				
	53.74	12.02				
Mean	53.84	12.03				
B. F. Robertson, South Carolina	54.08	12.14				
M. G. Donk, Florida	53.90	11.70				
	53.90	11.70				
	53.92	11.66				
	53.88	11.66				
Mean	53.90	11.68				
Geo. Roberts, Kentucky[c]	54.08	12.10				
	54.00	12.08				
Mean	54.04	12.09				
F. B. Carpenter, Richmond, Va	54.05	12.08	5.02	2.90		
Geo. A. Hanvey, Richmond, Va	54.05	11.98	5.12	2.94	5.30	3.06
C. Glaser, Baltimore, Md	[a] 53.13	[a] 12.67				
G. W. Lehman, Baltimore, Md	[a] [b] 53.99	[a] 11.18				
	[a] [b] 53.90	[a] 11.08				
	[a] 52.88					
	[a] 53.04					
General average	54.02	12.04				

[a] Omitted from general average. [b] Not washed with NH_4Cl. [c] Comments by A. M. Peter.

TABLE II.—*Results of determination of potash in samples.*

Analyst.	Lindo-Gladding method.						
	Sample No. 3.	Sample No. 4.	Sample No. 5.	Sample No. 6.	A.	B.	C.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
H. B. McDonnell, Maryland ...	5.01	2.86	5.25	2.97	0.06	0.25	0.69
	[a]5.05	[a]2.73	[a]5.19	[a]3.09	[a] .08	[a].26	[a].67
Mean	5.03	2.80	5.22	3.03	.07	.26	.68
T. R. Gough, Maryland	5.32	3.16	5.79	3.28	.20	.21	.58
	5.38	3.21	5.72	3.27	.24	.21	.56
Mean	5.35	3.19	[b]5.76	3.28	.22	.21	.55
H. D. Haskins, Massachusetts..	[b]4.78	2.72	4.98	2.74	....	....	....
M. H. Pingree, Pennsylvania ..	5.06	3.05	[c]5.31	[d]3.05	....	....	....
	5.08	2.99	[c]5.31	[d]3.00	....	....	....
Mean	[e]5.07	3.02	[c]5.31	3.03	....	....	....
John S. Burd, U. S. Dept. Agr...	5.08	2.83	5.15	3.04	....	....	....
	[a]5.14	[a]2.93	[a]5.19	[a]3.05	....	....	....
Mean	5.11	2.88	5.17	3.05	....	....	....
B. F. Robertson, South Carolina	5.26	2.93	5.33	2.99	....	....	....
M. G. Donk, Florida	5.01	2.73	5.24	2.93	....	....	....
	5.06	2.76	5.27	2.95	....	....	....
	5.02	2.60	5.27	3.01	....	....	....
	4.99	2.54	5.28	3.03	....	....	....
Mean	5.02	2.66	5.27	2.98	....	....	....
Geo. Roberts, Kentucky[f]	5.03	2.90	5.22	3.07	....	....	....
	5.04	2.75	5.26	3.05	....	....	....
Mean	5.04	2.83	5.24	3.06	....	....	....
F. B. Carpenter, Richmond, Va.	5.01	2.70	....	....	....	....	....
G. A. Hanvey, Richmond, Va ..	5.09	2.77	5.17	2.97	....	....	....
G. W. Lehman, Baltimore, Md .	4.87	2.64	5.13	2.73	....	....	....
	4.90	2.69	5.15	2.81	....	....	....
	4.99	....	....	....	....	....	....
	4.95	....	....	....	....	....	....
Mean	4.93	2.67	5.14	2.77	....	....	....
Average	5.09	2.83	5.20	2.99	....	....	....
Theory	5.46	3.06	5.62	3.19	....	....	....

[a] 250-cc flask.

[b] Omitted from average.

[c] Ignition with H_2SO_4 and solution in water, 5.49 and 5.47.

[d] Ignition with H_2SO_4 and solution in water, 3.25 and 3.09.

[e] Used analyst's acid phosphate.

[f] Comments by A. M. Peter.

TABLE III.—*Results of determination of potash in samples.*

Analyst.	BaCl$_2$–Am$_2$CO$_3$ method.				Milk-of-lime method.			
	Sample No. 3.	Sample No. 4.	Sample No. 5.	Sample No. 6.	Sample No. 3.	Sample No. 4.	Sample No. 5.	Sample No. 6.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
H. B. McDonnell, Maryland	5.02	2.82	5.27	3.03	5.04	2.91	....	....
	[a] 5.12	[a] 2.82	[a] 5.31	[a] 3.13	[a] 5.03	[a] 2.93	....	....
Mean	5.07	2.82	5.27	3.08	5.04	2.92	....	....
T. R. Gough, Maryland	[a] 5.18	[a] 3.14	[a] 4.92	[a] 2.87	[a] 5.28	[a] 2.90	[a] 6.40	[a] 3.75
	[a] 4.83	[a] 2.77	4.97	2.75	5.24	2.88	6.21	3.63
	4.91	2.70	[a] 5.17	2.95	....	....	....	....
	....	2.70	5.27	....	....	....	....	....
Mean	4.97	2.82	5.08	2.85	5.26	2.89	6.31	3.69
H. D. Haskins, Massachusetts	4.98	2.86	5.27	2.95	4.77	2.73	5.99	3.65
M. H. Pingree, Pennsylvania	5.02	3.08	5.25	....	1.97	2.99	5.22	3.15
	[a] 5.14	[a] 3.01	[a] 5.38	....	[a] 5.03	[a] 2.96	[a] 5.20	[a] 3.02
Mean	[a] 5.08	[a] 3.05	[a] 5.32	[a] 3.09	[a] 5.00	[a] 2.98	[a] 5.21	[a] 3.09
John S. Burd, United States Department of Agriculture	4.97	2.75	5.39	2.77	....	....	....	....
	[a] 4.92	[a] 2.73	[a] 5.20	[a] 2.83	....	....	....	....
Mean	4.95	2.75	5.30	2.80	....	....	....	....
B. F. Robertson, South Carolina	5.28	3.04	5.31	3.01	5.33	3.01	....	....
M. G. Donk, Florida	5.11	2.71	5.22	2.96	4.88	2.72	[b] 5.48	[b] 3.02
	....	....	....	....	4.90	2.75	[b] 5.50	[b] 3.08
Mean	....	....	....	....	4.89	2.74	5.49	3.05
G. A. Hanvey, Richmond, Va	4.93	2.84	5.08	3.06	5.06	2.95	....	....
Average	5.04	2.87	5.23	2.97	5.05	2.88	5.75	3.37
Theory	5.46	3.06	5.62	3.19	5.46	3.06	5.62	3.19

[a] 250-cc flask. [b] Organic matter destroyed before making solution.

TABLE IV.—*Moisture in potash salts.*

Analyst and method.	1.	2.	9806 (Md., com.), KCl.	9807 (Md., com.), K$_2$SO$_4$.	9818 (Md., com.), KCl.	9975 (Md.), kainit.	10275 (Md., com.), NaCl and KCl, 12 per cent K$_2$O.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
C. G. Church, Maryland: combustion tube and CaCl$_2$	0.05	12.84	1.91	0.81	3.15	11.65	3.28
	.05	12.64	1.70	.98	3.19	11.88	3.33
T. R. Gough, Maryland:							
100° C	.01	1.71	.84	.32	1.80	1.40	2.73
	.00	1.74	.90	.31	1.85	1.43	2.68
130° C	.00	5.60	1.60	.73	2.97	5.37	3.00
	.00	5.65	1.59	.73	2.95	5.26	3.08
Red heat	.09	13.20	1.61	1.10	3.35	11.81	3.35
	.09	13.15	1.65	1.10	3.20	11.90	3.33
M. G. Donk, Florida:							
Official	.00	4.24	....	....	....	....	....
	.00	4.27	....	....	....	....	....
Ignition	.07	14.68	....	....	....	....	....

TABLE IV.—*Moisture in potash salts*—Continued.

Analyst and method.	1.	2.	9806 (Md., com.), KCl.	9807 (Md., com.), K_2SO_4.	9818 (Md., com.), KCl.	9975 (Md.), kainit.	10275 (Md., com.), NaCl and KCl, 12 per cent K_2O.
C. Glaser, Baltimore, Md.:	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
100° C	0.03	2.63					
Ignition on hot plate	.09	12.17					
G. W. Lehman, Baltimore, Md.:							
130° C	.00	7.37					
Dull red heat	.08	14.31					

COMMENTS OF ANALYSTS.

H. D. Haskins, Massachusetts.—Ten grams to 500 cc were used for the tests; for the duplicates 5 grams to 250 cc were used. In the last set of determinations, by the new or last method, duplicates were not run on account of there being an insufficient amount of the acid phosphate or bottle A.

M. H. Pingree, Pennsylvania.—The first results were obtained from solutions of 10 grams made up to 500 cc; the second represent solutions of 5 grams made up to 250 cc. The amount of acid phosphate sent by referee being too small to complete the work, a similar sample found to contain 0.10 per cent K_2O was substituted, as noted in Table III (foot-note *e*). The samples of kainit, fish scrap, and dried blood, represented by the number 2 and the letters C and B, were very coarse and gave me considerable difficulty in obtaining uniform samples.

John S. Burd, United States Department of Agriculture.—You will observe the absence of results by the "lime method," and I have to report lack of success with same in that I was unable to dissolve the excessive quantities of lime salts by the use of the ammonium chlorid washing mixture. Moisture in No. 1, official method, 0.01 per cent; by German method, 0.10 per cent. Moisture in No. 2, by official method: The sample did not attain constant weight after thirty-six hours at 130° C.; I therefore give the losses as recorded at six-hour intervals for that period: 6.46, 7.08, 8, 8.15, and 8.28 per cent. Moisture in No. 2, German method (with lime), 12.48 per cent.

A. M. Peter, Kentucky.—I am opposed to reducing the amount to be weighed out for potash in mixed fertilizers. Ten grams is none too large, and the use of a small quantity would tend to introduce irregularities from irregular distribution of the potash salts in the samples, even when carefully prepared. I believe it would be better to increase the amount of water used in boiling up the sample. In the case of potash salts the quantity weighed out might be reduced and the amount of water remain the same. This is our usual practice in this laboratory, and Mr. Roberts has followed it in his work. Allow me to suggest also that the two series of experiments which you propose for testing the relative merits of the use of 500 and 250-cc flasks will give you no data upon the real point at issue. When you are dealing with pure salts, as in these experiments, it is immaterial whether you weigh out 10 grams and make up to 500, or 5 grams and make up to 250 cc, but in a mixture the larger quantity is more likely to give a true result.

M. G. Donk, Florida.—Ten-gram charges were used in each case, as the hygroscopic nature of the kainit would not allow weighing of less than 2.5 grams with accuracy. The ignited residue, after evaporation with sulphuric acid was, with samples 3, 4, and 6, not perfectly soluble in hot water acidified with HCl, but a minute gelatinous residue had to be filtered off. The milk of lime method carried out according to directions on page 18(c) of last year's Proceedings[a] gave a pure K_2PtCl^6 precipitate with 3 and 4, but the dissolved organic matter in 5 and 6, on evaporation with platinum solution, left an impurity which no amount of washing would remove. The K_2PtCl_6 crystals also were colored deep brown by this same impurity. The results were so far out that I left them out of the table. Those in the table are results obtained by the method described on page 20 of last year's Proceedings.[a] In this case also, although the material was ignited to redness till all sulphuric acid had been volatilized, the same trouble was met with, though much less pronounced. The crystals in this case were apparently pure, only the mat of the Gooch being colored. After weighing the Gooch and precipitate, I dissolved the crystals with hot water and made a second weighing to correct for the organic matter present, but no increase in weight could be detected on account of the coloring matter.

F. B. Carpenter, Richmond, Va.—All of the results are considerably below what we would expect. With results marked "HCl" the Lindo-Gladding method was followed, with the exception that 5 cc of HCl was added to the solution before boiling. While the average of these results are a little higher than those obtained by other methods, there does not seem to be the difference that we usually have obtained in our previous experimental work with this modification. I trust some of the members of the association will be able to make some modification of the method now in use which will give theoretical percentages. As you will see, this is quite a serious question from the standpoint of the manufacturer.

G. W. Lehmann, Baltimore, Md.—On sample No. 1, when precipitate was washed with 95 per cent alcohol only, results were 53.99 and 53.90 per cent K_2O. When washed with 80 per cent alcohol and 25-cc portions of NH_4Cl solution, as in official method, results were 52.88 and 53.04 per cent. Determinations on No. 3 were made four times, because the figures found did not correspond with the calculations, and I am absolutely at a loss to account for the discrepancy.

C. G. Church, Maryland.—Moisture by combustion and absorption. In each case 10 grams of the sample were heated at a low red heat in a hard glass tube in an ordinary combustion furnace, and the moisture thus liberated, absorbed by passing it through a U tube containing $CaCl_2$. The tube was weighed before and after the combustion, and the difference calculated as moisture. The average time of combustion per sample was twenty-five minutes. Samples Nos. 2 and 9975 show such a large percentage that it was thought that some chlorin might have been absorbed. This was tested by passing dry chlorin through the $CaCl_2$ tube. In two experiments there was practically no increase in the weight of the tube. Three results were obtained on No. 2, but only two were concordant.

COMMENTS OF REFEREE.

The new method sent out, which is a modification of the alternate method, has been used by me occasionally for some years to check results by the Lindo-Gladding method, and has always given fairly concordant results. The ignition of the residue is free from the frying and sputtering that takes place in the latter method, which often causes us to fear that potash is lost in the fine spray of acid that is thrown up. The ignited residue is usually completely soluble, except where much organic matter is present.

The milk of lime method is rapid, till one comes to washing the final precipitate, where the large amount of lime, salts, etc. is troublesome. The method is, of course, not applicable to samples containing organic matter, like samples 5 and 6, or ammonium salts, unless such samples are first ignited, as has already been pointed out by the originators of the method. This limited application of the method is a very serious objection to it, especially in States that require the potash to be soluble in water.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 67.

The results further emphasize the fact that our methods fail to recover all of the potash from mixed fertilizers, especially those containing acid phosphate. The close agreement among all the methods certainly proves that the loss is due either to the fact that some of the potash forms an insoluble compound with silica or other substance present, or that it is "occluded" in the precipitate of phosphates, etc. Further work should be directed to the matter of getting the potash in solution. The low results are certainly independent of the size of the flask in which the solution is made. The theory that has been advanced that the potash salts are more liable to run in streaks in a sample than are the other ingredients, making it necessary to weigh out a much larger sample for that determination, I believe to be erroneous. Any irregularity in the potash salt surely will affect the nitrogen and phosphoric acid in the same ratio that it does the potash. Our concordant results on nitrogen with 1-gram charges and phosphoric acid with 2-gram charges certainly is sufficient argument against such a theory.

A copy of the method of analyzing potash salts, as practiced by the German chemists, was kindly furnished by Mr. von Herff. Two salient features of the methods are the weighing out of a charge of such size that the final weight of the precipitate will show the percentage without calculation, and also the fact that they change the potash to the form of chlorid, by precipitating the sulphuric acid by barium chlorid, before adding the platinum solution.

To get the theoretical percentage of potash, as given in the tables, the averages of results on samples 1 and 2 were taken, to which was added the amount of potash derived from the other ingredients, as shown by the analyses of the referee. The average results of the different methods agree very closely, and, averaging all results, the average loss of potash in Nos. 3 and 4, in which 10 per cent of sulphate of potash was used, was 0.40 per cent, or a little over 7 per cent of the total amount present. The average loss in samples Nos. 4 and 6, containing 25 per cent of kainit, is 0.20 per cent, or slightly less than 7 per cent of the total amount present.

While the determination of moisture is of minor importance to the fertilizer inspector, the important one being the content of the valuable constituents, it is desirable that our methods in this respect give accurate results. Probably this may be attained in the case of salts, though it will certainly be a difficult problem in the case of mixtures of organic compounds with potash compounds like kainit. The combustion method, absorbing the water in calcium chlorid, is easy of application, and I think will give good results, but needs further trial.

For recovering waste platinum, I prefer to keep the precipitates and filtrates separate. The precipitates are treated with hot water and the asbestos separated by filtration, the filtrate being acidified with hydrochloric acid and a little pure granulated zinc added to effect the reduction.

The alcohol filtrate is concentrated by evaporation and treated in the same way. The ammonium chlorid filtrate, which is caught in another filter bottle, is thrown away, as the amount of platinum in it is very minute.

RECOMMENDATIONS.

The referee recommends:

(1) That further experiments be made for the purpose of determining a method that will dissolve all of the potash in mixed fertilizers.

(2) That further experiments be conducted with methods for determining moisture in potash salts.

(3) That the method of analysis shall give some latitude for the judgment of the analyst in regard to the amount of substance taken.

The **President.** You have heard the report on potash. It has been announced that one paper on this subject is to be presented, but as it is not ready at this time it will be presented later.

Mr. Donk. I wished to present a paper on this subject, but did not have time to write a formal report and include the calculations. I would like, however, to say a few words as to the probable cause of the low potash results obtained by our present methods with mixtures containing acid phosphate.

LOW POTASH RESULTS OBTAINED BY THE PRESENT METHOD WITH MIXTURES CONTAINING ACID PHOSPHATE.

By M. G. Donk.

Working on the comparison of methods for estimating potash in mixed fertilizers, carried out according to the instructions of the referee, Mr. McDonnell, it was not possible with any of the three methods compared to recover all the potash added in preparing the samples. This is in harmony with the results of last year's work, one of the objects of which was to see if it is possible with the Lindo-Gladding method to obtain from mixtures of acid phosphate and potash salts the theoretical amount of potash added. The potash work for 1900 also brought out the fact that the Lindo-Gladding method fails to recover all the potash in mixtures of potash and acid phosphates. In 1900 the loss was 3.92 and 4.95 per cent, respectively, with the two samples used, and in 1901 the loss was 6.86 per cent, while my results for this year's work are no better. Thinking that the loss was occasioned by the potash being carried down by the precipitate of interfering substances, I performed the following experiment:

Sample No. 1: Potassium sulphate, 0.1 gram, and acid phosphate, 0.9 gram.

Sample No. 2: Kainit, 0.25 gram, and acid phosphate, 0.75 gram. Added 30 cc of water and boiled for thirty minutes; filtered off the insoluble residue; washed and evaporated the filtrate down to 30 cc, when I removed lime, etc., by the Lindo-Gladding method. The precipitate I washed until free from chlorin and sulphate; dried at 100° C.; pulverized and again washed until free from sulphate and chlorin. Dissolved the precipitate with smallest possible quantity of hydrochloric acid, washed and diluted filtrate to 50 cc; then reprecipitated by addition of ammonia and a few drops of ammonium oxalate; filtered, washed, and determined potash in filtrate by Lindo-Gladding method. Sample No. 1 gave 0.029 per cent K_2O; sample No. 2, 0.071 per cent K_2O. The early meeting of the association prevented my experimenting along this line further, as was my intention, and a single test gives but meager data on which to base an opinion. Yet so far as it goes it tends to show that potash is occluded by the precipitate formed in the removal of interfering bodies in all the methods thus far tried. And this is to be expected from the nature of the process, for it is a well-known fact in chemistry that a chemically pure precipitate can but rarely be obtained from a solution containing other salts than the one it is desired to precipitate. Further, the more concentrated the solution the greater the contamination of the precipitate by the material in solution, and it is for this reason that I agree with Mr. Huston, who has found, as stated at the seventeenth annual meeting (see Proceedings, pp. 15 and 16), that the longer certain materials are heated the smaller the amount of potash found, owing to concentration of the potash solution from which the interfering substances must be precipitated. For the same reason a higher percentage is obtained by his method of passing steam through the solution instead of boiling, as no concentration is thus possible, while an increase in the volume of potash solution is brought about. This evil of the precipitate removing part of the potash from the solution is inherent in every method thus far tried by the association. I was therefore much interested in a method first brought to my notice by Dr. Herzog, of Gainesville, Fla., in which it is possible to remove the potash from the solution in place of the interfering salts. This may be done by precipitating the potash as potassium phosphomolybdate in acid solution. The possibilities of this method have not been sufficiently tested to decide as to its accuracy, but it appears to me that by properly studying the conditions influencing the precipitation, as has been done for the optional volumetric method for phosphoric acid, it may be developed into a method by which it is possible to recover all the potash from such materials as make up the mixed fertilizers now on the market.

I wish to point out again the want of experimental evidence as to the statements first made, and would not have presented this paper but for the fact that it may suggest to others investigations along this line. In conclusion, I wish to say that if there are any members present who are in a position to offer any suggestions, information, or criticism on this proposed method I would be very glad for them to do so, as I am going to study it during the coming year.

The PRESIDENT. Is there anything to be said in response to this request?

Mr. CAVANAUGH. I may be able to answer the gentleman who has just spoken on the question of potash. The graduate school of our chemical department is working on that subject, and after one year reports very favorable results on the method last mentioned, carrying it on as a volumetric method. The results are agreeing better and giving more satisfaction than those obtained by the Lindo-Gladding method.

Mr. JAFFA. Mr. President, the committee to wait upon the Secretary and Assistant Secretary of Agriculture; and President Needham, of Columbian University, is ready to report, as follows: The president of the Columbian University said he would be glad to attend one of our sessions. We met the Secretary of Agriculture on our way, and he said he would be pleased to accept the invitation. The Assistant Secretary is out of town, but may return by Saturday morning. We will then call and extend the invitation.

Mr. DAVIDSON. I move that we adjourn until 1.30 p. m.

The motion prevailed, and the meeting adjourned at 12 o'clock noon.

THURSDAY—AFTERNOON SESSION.

The meeting was called to order at 1.30 p. m.

The PRESIDENT. The referee on foods and feeding stuffs has requested that he be allowed to defer his report until to-morrow morning. The next in order will be the report on dairy products.

Mr. CAVANAUGH. The report on dairy products will be a very short one. It will be only an attempt to throw a little light on the difficulties that have been experienced in the past.

REPORT ON DAIRY PRODUCTS.

By G. W. Cavanaugh, *Referee.*

In 1897 Dr. Van Slyke, as referee on dairy products, reported results on the determination of albumin in cow's milk. Three methods were tried. These same methods, with slight modifications, were again tried in 1898 and 1899. In 1900 and 1901 two more methods were added. The results of the coworkers have never been satisfactory. The following table shows highest and lowest results reported:

Table I.—*Determination of albumin in cow's milk.*

Method.	1897.		1898.		1899.		1900.		1901.	
	Minimum.	Maximum.	Minimum.	Maximum.	Minimum.	Maximum.	Minimum.	Maximum.	Minimum.	Maximum.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1	0.251	0.370	0.560	0.660	0.350	0.554	0.140	0.380	0.210	0.328
2	.175	.431	.390	.460	.460	.564	.100	.450	.240	.345
3	.373	....	.440	.600	....	....	.310	.530	.270	.375
4	....	....	....	....	....	....	.350	.610	.210	.312
5	....	....	....	....	....	....	.020	.610	.280	.312

During these five years the samples of milk were preserved with formalin. Van Slyke mentions the proportion, 1 cc of a 40 per cent solution per liter. His report shows it to be a good preservative for milk when casein is to be determined, but no figures were given for albumin. No mention is made by the other referees of the amount of formalin used.

On page 114 of the Proceedings for 1901[a] I find the following resolution:

Resolved, That it is the sense of the association that until a proposed new method or modification of an existing method has been tested by the referee with favorable results it shall not be submitted by him for general test by this association.

Looking over the results of the last five years, the referee decided to try the methods before sending out samples. The present official method was first tested on a sample of fresh whole milk, and one of fresh separated milk. Samples for duplicate determinations were removed and then formalin was added to preserve the remainder. The following table shows the amounts of albumin found during a period of four days:

		Per cent.
First day	a. m.	0.525
	p. m.	.508
Second day	a. m.	.463
	p. m.	.376
Third day		.358
Fourth day		.157

Mr. B. B. Turner, Ph. D., using the official method on a fresh sample found 0.479 per cent and after three days, with the sample preserved with formalin, 0.122 per cent. These few figures are only a sample of the discouraging results obtained in the referee's laboratory, In looking for a possible explanation the following facts are observed: The existing official and provisional methods give satisfactory concordant results when tried on fresh milks. The same methods in the hands of chemists who have reported to the referees during the last five years give very unsatisfactory results. The milks were all preserved with formalin. In 1900 the lowest figure was reported from Oregon to the referee in New York. This agrees with the referee's figures given above.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 67.

In the Zeitschrift für Physiologische Chemie,[a] Dr. F. Blum calls attention to a new class of compounds of albumin in the following words:

> A few drops of formalin added to a solution of egg albumin prevents the coagulation of the albumin by heat even after the solution is concentrated and the formalin driven off.

It needs, of course, demonstration that a similar effect is produced on the albumin of milk, before this offered explanation has much weight. To test this, four portions of milk of 10 cc were set aside for two days with respectively 2, 4, 6, and 8 drops of formalin. They were then diluted with 90 cc of water and the official method was carried out as for albumin. The portions having the smallest amounts of formalin showed a slight precipitation on boiling. No. 3 gave but a faint opalescence, while No. 4 remained perfectly clear even after forty-five minutes' boiling.

I recognize that the preservative was added in proportions greatly in excess of that necessary to preserve milk. As this preservative is known to prevent the coagulation of egg albumin, it seems quite probable that a like effect may occur with milk albumin. For this reason the referee did not send out samples to other laboratories.

The President. We will now listen to the paper on potash to be presented by Mr. Ross.

Mr. Ross. I shall give a brief synopsis of the paper submitted by Mr. Hare:

THE DETERMINATION OF POTASH IN FERTILIZERS BY SUBSTITUTING MILK OF LIME FOR AMMONIA AND AMMONIUM OXALATE AS THE PRECIPITANT.

By C. L. Hare.

The Lindo-Gladding method for the determination of potash in fertilizers possesses one particularly objectionable feature: The evaporation and ignition of an aliquot with sulphuric acid for the purpose of expelling ammonia salts and destroying dissolved organic matter is a tedious process and involves probable loss from sputtering during evaporation. A reliable modification which would exclude this feature would be highly desirable. At the sixteenth annual meeting of the Association of Official Agricultural Chemists Mr. Ross presented a method based upon the use of barium carbonate as a precipitant for phosphates, iron, alumina, etc. He later suggested milk of lime as the precipitant, and the writer, in 1901, reported results obtained by collaborators on official methods for potash determination. The method employed is as follows:

> Weigh out 10 grams of the sample and boil with about 350 cc of water for thirty minutes; while hot add milk of lime till slightly alkaline. Cool, make up to 500 cc with water, and remove 50 cc, corresponding to 1 gram. Acidify with hydrochloric acid, add platinum solution, and evaporate to soft dryness over water bath. The residue is then washed with 80 per cent alcohol and ammonium chlorid "wash," as in the Lindo-Gladding method.

In the case of fertilizers containing organic matter, treat 10 grams of sample with dilute sulphuric acid (1-1), and incinerate; moisten ignited mass with dilute sulphuric acid (1-1), and heat to aid in removing from the dish; boil with 350 cc water and proceed as before. The milk of lime precipitates all lime except that present as sulphate, and the solubility of the sulphate being only 1 in 500, the aliquot of 50 cc will contain only 0.1 gram of that substance, an amount readily removed by the ammonium chlorid wash water and insufficient to cause material trouble either when solution is made with water direct or after incineration with sulphuric acid.

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[a] See vol. 22, p. 127.

The potassium platinic chlorid crystallizes well, and, after washing with alcohol, the salts of lime appear in light flakes which require never more than six washings with ammonium chlorid "wash" for complete removal.

The modification possesses several advantages over the Lindo-Gladding method: (1) The precipitation of lime is immediate, and there is no delay at this point; (2) evaporation to expel ammonia salts is eliminated; (3) probable loss of potash during evaporation is obviated; (4) washing the potassium platinic chlorid with alcohol and ammonium chlorid "wash" may be performed thoroughly with greater ease and rapidity, while no larger amount of platinum is required than by the Lindo-Gladding method.

The accuracy of the method is evidenced by results as shown in the tables. It has been used by the writer upon several hundred samples of miscellaneous fertilizers as a check against the Lindo-Gladding, and it is evident that the modification would not be applicable to fertilizers containing ammonia salts.

Table I gives parallel results by the milk-of-lime and the Lindo-Gladding methods upon fertilizers containing no organic materials, solution in each case being made by boiling the sample with water:

TABLE I.—*Determination of potash in fertilizers containing no organic materials.* [a]

Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1.02	1.29	1.66	1.88	4.11	4.12	1.95	1.66
1.42	1.62	1.76	1.88	1.06	1.29	3.91	3.86
4.51	4.53	2.59	2.44	3.91	4.02	1.21	1.41
2.33	2.20	1.40	1.65	1.96	1.89	1.50	1.80
1.91	1.91	.84	.80	2.59	2.63	1.49	1.32
1.97	1.87	4.33	4.53	2.46	2.48	5.33	5.65
2.42	2.42	2.44	2.35	4.36	4.47	2.16	2.00
1.95	2.12	2.41	2.35	3.12	2.93	4.26	1.10
2.54	2.64	5.24	5.17	2.10	2.08	2.72	2.76
2.56	2.45	1.58	1.73	2.52	2.26	2.49	2.76
2.44	2.36	2.29	2.08	2.11	2.27	3.41	3.40
1.09	1.04	.81	.98	2.50	2.76	2.51	2.67
1.10	.90	2.37	2.29	4.37	4.33	4.21	4.04
2.14	1.89	3.66	3.90	2.49	2.74	2.77	2.41
1.75	1.89	2.44	2.88	1.75	1.89	2.01	1.88

[a] Solution by boiling sample with water.

Inspection of the tables reveals differences ranging from 0.01 to 0.27 per cent, with two exceptions, in a total of 60 samples. Of this number, 46 show differences less than 0.20 per cent, while the average difference is 0.10 per cent.

This average difference is, of course, without value as an indication of accuracy, unless considered in connection with the variation of each parallel determination, but when so large a percentage of duplicates falls within the limits of probable error the average in differences of the entire number of determinations indicates in a measure the comparative accuracy of the two methods.

Considering that the figures in the tables represent first results, and in no case a second determination by the same method, results by the milk-of-lime method appear quite as reliable as those obtained by the Lindo-Gladding method. In the regular course of laboratory work duplicate determinations by one and the same method may easily show less closely agreeing results.

Table No. II shows parallel results obtained from fertilizers containing organic materials, the samples in each case being treated with dilute (1-1) sulphuric acid and incinerated before making solution in water:

TABLE II.—*Determination of potash in fertilizers containing organic materials.* [a]

Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
2.16	2.21	2.37	2.60	1.59	1.57	2.44	2.63
1.87	1.84	1.92	1.96	2.73	2.59	2.20	2.29
2.58	2.53	1.66	1.70	1.32	1.46	3.22	3.11
1.71	1.70	2.59	2.45	2.37	2.10	2.21	2.13
1.31	1.51	2.33	2.38	2.71	2.61	1.31	1.31
2.13	2.24	2.09	2.11	2.65	2.53	2.41	2.28
2.27	2.39	2.12	1.96	2.16	2.20	2.28	2.33
2.46	2.48	3.46	3.33	2.61	2.89	3.67	3.46
1.48	1.49	2.41	1.93	1.62	1.82	3.38	3.36
1.35	1.08	2.47	2.50	2.66	2.42	.74	.84
1.59	1.60	2.46	2.73	2.38	2.08	2.34	2.22
2.07	1.80	4.84	4.69	1.97	1.78	2.69	2.60
1.51	1.50	2.53	2.57	1.16	1.20	2.43	2.44
2.49	2.38						

[a] Samples treated with dilute sulphuric acid and incinerated prior to solution in water.

By reference to Table II we find variations ranging from 0.01 to 0.48 per cent in a total of 53 determinations. Forty-one of these give differences of 0.20 per cent or less, results averaging 0.06 per cent higher by the lime method than by the Lindo-Gladding.

Table III contains results from fertilizers containing organic materials. Samples were ignited with sulphuric acid (1-1) before obtaining solutions for the lime method, while for the Lindo-Gladding, solution was obtained by boiling the unignited sample with water.

TABLE III.—*Determination of potash in fertilizers containing organic materials.* [a]

Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1.62	1.66	1.92	2.11	4.00	3.87	2.88	2.60
2.02	2.19	2.58	2.74	.50	.69	2.24	2.21
2.14	2.11	2.38	2.23	2.18	2.47	2.97	2.94
.88	.74	1.92	2.09	1.18	1.20	1.43	1.26
1.11	1.06	2.71	2.64	1.96	1.73	1.95	2.06
1.82	1.55	1.56	1.34	1.78	1.68	1.84	2.14
1.53	1.68	1.98	1.96	2.00	2.07	1.93	2.14
1.61	1.63	1.15	1,28	2.09	2.35	2.37	2.56
3.09	3.02	2.28	2.54	1.52	1.62	1.65	1.81
1.84	2.06	1.29	1.53	1.80	1.78	3.70	3.46
2.05	2.10	2.53	2.29	2.17	2.25	3.41	3.52
2.43	2.32	1.32	1.28	1.62	1.70	1.54	1.30
2.52	2.65	2.26	2.07	1.65	1.71	1.35	1.48
2.95	2.86	1.50	1.49	2.38	2.43	2.68	2.80
2.00	2.29	1.88	1.60	1.83	1.64	2.85	3.13
1.95	2.03	2.94	3.00	2.63	2.37	3.03	3.02

[a] Lime method: Samples ignited before solution. Lindo-Gladding method: Unignited sample boiled with water.

These results show an extreme variation of 0.29 per cent in a total of 63 determinations. Forty-six of the 63 vary less than 0.20 per cent, while the results average 0.02 per cent lower by the lime method.

In Table IV may be found results on fertilizers containing organic materials, solutions of both methods being obtained by boiling the sample with water. The solutions for the lime method were considerably colored by dissolved organic matter.

TABLE IV.—*Determination of potash in fertilizers containing organic materials.* [a]

Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.	Lime method.	Lindo-Gladding method.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1.27	1.21	1.43	1.08	2.50	2.05	2.17	2.24
1.04	1.15	1.79	1.71	2.69	2.31	5.85	5.56
2.71	2.61	5.51	5.51	2.15	2.18	2.16	1.87
1.34	1.16	5.03	4.61	1.54	1.17	2.30	1.83
1.74	2.06	2.01	1.92	1.95	2.04	2.43	2.09
1.38	1.21	2.48	2.18	2.57	2.35	2.40	2.25
2.87	2.44	2.70	2.20	2.50	2.24	1.00	1.10
2.65	2.35	2.64	2.40	2.19	2.14	2.41	1.93
2.28	1.83	3.42	3.38	2.11	2.11	2.30	2.10
2.50	2.54	2.53	2.42	1.16	1.20	....	....
1.28	1.25	2.23	1.80	2.43	2.45	....	....
2.41	2.11	2.35	2.49	1.29	.99	....	....

[a] Samples boiled with water.

Although little was expected from the results obtained by the lime method under these conditions, still they show an extreme variation of only 0.55 per cent in a total of 45 determinations, while 30 of the 45 differ less than 0.30 per cent and the average difference is only 0.15 per cent. Nothing is claimed for the method when used under these conditions, but it is interesting to note that the method gives many good results, even when precipitation of platinum takes place in a solution considerably colored with organic matter.

Mr. **CRAMPTON**. Mr. President, I would like to say that if any member of the association knows of a test for the presence of palm oil in small quantities, less than 1 per cent, in mixtures of other fats (oleomargarine) and will communicate with me direct he will confer a great favor. Such a test is very much needed in the administration of the new internal-revenue law taxing artificially colored oleomargarine.

Mr. **JAFFA**. I would like to say that an invitation has been extended to Dr. Munroe to attend this meeting. He thanks the association for the invitation and will visit us to-morrow morning. I believe this completes our report.

Mr. **DAVIDSON**. Mr. President, I have been informed that the Census Bureau has published a bulletin entitled "Chemicals and Allied Products," prepared by Dr. Munroe, which would be of great interest to those who have not seen it. Dr. Munroe says that he, himself, can not furnish a sufficient number of copies to supply all the members of the association, and suggests that if the association should request copies for all the members they could probably be secured. I would like to move that the secretary of this association be instructed to request Dr. Munroe to procure a sufficient number of copies of this bulletin to supply all the members with one.

Mr. McDonnell. I am quite sure, Mr. President, that all the members would be glad to secure a copy of this publication.

The motion prevailed.

The President. Has the referee on insecticides arrived?

Mr. Haywood. I have sent out for my report and it will be here in the next few minutes. I had not expected that it would come up this afternoon. In the meanwhile, I have a short communication here which was received from Mr. Gudeman, of Chicago, by Dr. Wiley, who asks me to read it. It is not on insecticides, but on feeding stuffs.

The President. If there is no objection, we will listen to the paper to be offered on feeding stuffs.

GLUTEN FEEDS: DETERMINATION OF FAT AND ACIDITY.

By Edward Gudeman, *Chicago, Ill.*

The extensive use of the maize gluten feeds, manufactured in starch and glucose factories, is a comparatively new thing. These feeds have been on the market about twenty years, but the present method of manufacture dates back only about five years. A large amount of work has been done and is being done on this class of feeds, especially by the agricultural experiment stations. The data published as to these feeds goes back to 1883 and comes up to 1900, and while mainly correct does not fully apply to the feeds as manufactured to-day. In many cases, as no distinction is made between the different feeds, the data is misleading.

The starch and glucose works of the United States have a daily capacity of about 225,000 bushels of corn. Of this quantity about 15 pounds per bushel is obtained in the form of cattle feeds. The yearly production is about 500,000 tons, 75 to 85 per cent of which is consumed in the United States, the remainder being exported. The exports are confined mainly to the gluten meal and the corn oil cake. The factories are located in the East and West, and depend on the whole corn belt for their raw material, about 68,000,000 bushels per year, which amounts to about 3 per cent of the average annual corn crop of the United States.

Corn varies in composition and it would be natural to expect these feeds to vary accordingly. This is not the case, and in a well regulated factory the feeds run very uniformly, much more so than wheat bran, cornmeal, or any of the common cattle feeds made from raw cereals. The kernel of corn is divided into its component parts, and while the proportions of these vary the parts themselves remain practically constant. As the process of manufacture of these feeds has a direct bearing on the analytical results, a brief superficial outline of the same is necessary.

Shelled corn is soaked in warm water thirty-six to sixty hours. This water contains a small amount of sulphurous acid and about 8 gallons of it is required per bushel. The corn swells and absorbs about one-half of this acid water. After soaking the required time, the sulphur water is drawn off, and as this contains a large amount of soluble substance of high feeding and fertilizing value, it is concentrated and mixed with the feeds in the same proportion as it is obtained from the corn. Analyses of the dry substances contained in this extract will show 6 to 10 per cent salts and 15 to 30 per cent of protein (N×6¼). Sulphurous acid acts peculiarly on the kernel, penetrating strongly into the same and quickly loosening the intercellular tissue, thereby causing a good separation of the starch from the gluten at a later stage of the process. It also acts as a preservative, preventing to some extent the fermentation of the grain.

The soft wet corn is very coarsely ground in such a way that the germ is separated without being broken, cut, or crushed. This mass is made up to a certain specific gravity with starch milk and placed in suitable tanks. The heavy material will sink to the bottom while the light oily germs float. These tanks are so arranged that this separation is a continuous and automatic one. The germs are washed free from all adhering starch, pressed and run into driers with about 50 per cent of water from which they are discharged with less than 3 per cent of water. The dry germs are now treated exactly as linseed; ground, cooked, steamed, and subjected to hydraulic pressure, giving the corn oil and the corn oil cake, the latter, if ground, being known as germ oil meal.

The mass that sinks to the bottom of these tanks is reground to a very fine pulp and thoroughly washed on bolting-cloth sieves. The starch and the gluten pass through the sieve and the coarse material (the bran) over the end of the sieve. This bran is pressed and goes to the driers with 50 to 60 per cent water, comes out of them with about 12 per cent, and is sold as corn bran or under some fancy name. To separate the germs and the bran from the starch and the gluten, about 60 gallons of fresh water are used per bushel of corn. All of this water is run into tanks for concentration and recovery of the starch and gluten. After concentration it is again diluted with about 60 gallons of fresh water per bushel of corn and run on the starch tables, where the starch deposits and the gluten flows off into another series of concentrating tanks, where it is allowed to settle before going to the presses. The pressed gluten goes to the driers with 50 to 60 per cent water and is dried down to about 12 per cent, which gives the gluten meal of commerce. A mixture of gluten meal and corn bran in the same proportion as obtained from the corn is the gluten feed of commerce. Generally this mixture is made wet before the bran or gluten has been pressed.

All the waters separated in these various tanks go directly to the sewer, carrying along all soluble impurities and the sulphurous acid absorbed by the corn in the soaking process. All in all about 120 to 130 gallons of fresh water are used to a bushel of corn to wash out the starch from the germ, bran, and gluten. As the feeds and germs contain about 50 per cent of water as they go into the driers and the same percentage is in the starch separated on the tables, we have 110 to 120 gallons of water per bushel that have been discharged into the sewer, and it is self-evident that very little of the acid used for soaking the corn will remain in the feeds. The small amount that is found in these feeds comes from the concentrated soak water that is added to the feeds after they have been pressed. The larger percentage of this is lost during the drying of the feeds. The concentration of this soak water in vacuum pans takes out fully 90 per cent of the sulphurous acid before the same is added to the feeds.

The old process, now almost completely discarded, made use of alkali to separate the starch from the gluten. This gluten had to be acidified before it could be economically handled. Sulphuric acid was commonly used, quite in excess, to make the gluten settle. This settled gluten was pressed or mixed with bran and then without any further washing went to the driers. Such feeds were high in acid, and there are records of cattle having become mouth-sore owing to the excessive amount of sulphuric acid in these feeds.

The methods of the Association of Official Agricultural Chemists are the standards used in the United States for making feed analyses. The methods for the determinations of water, ash, fiber, and crude protein ($N\times6\frac{1}{4}$) give satisfactory results. The method for the determination of fat, applied to this class of feeds, is open to criticism. For the determination of acidity no official method exists, and none of the methods known to me give like results.

The official method for the fat determination says: "Extract the substance dried as for the determination of moisture." This means drying the substance in hydrogen or vacuum for five hours. As a general rule the drying is prolonged from eight to twenty-four hours. Drying the gluten feeds in hydrogen, vacuum, or air modifies the fat materially, besides changing the character of other constituents. The fat is made partly insoluble in ether or carbon-disulphid. The error due to this change in the solubility of the fat is often sufficient to class a feed below its guaranteed standard if the fat is just up to this standard.

TABLE I.—*Percentages of fat in gluten feeds.*

Samples and preparation.	Extracted with—	Forms of feed sampled.			
		Gluten meal.	Gluten feed.	Corn bran.	Germs.
Samples A:		*P. ct. fat.*	*P. ct. fat.*	*P. ct. fat.*	*P. ct. fat.*
Without drying	CS_2	4.74	3.02	3.97	
Dried over H_2SO_4	do	4.72	3.05	3.97	
Dried in hydrogen	do	3.60	2.61	3.74	
Dried in vacuum	do	3.62	2.61	3.75	
Dried in air	do	3.60	2.62	3.75	
Samples B:					
Dried in hydrogen	Ether	3.55	2.67	3.83	
Dried in hydrogen	CS_2	3.61	2.67	3.81	
Dried in air	do	3.58	2.68	3.80	
Not dried	Ether	4.68	3.00	3.93	
Not dried	CS_2	4.66	3.01	3.96	
Samples C:					
Not dried	Ether	5.16	6.19	5.32	
Not dried	CS_2	5.14	6.21	5.32	
Dried in hydrogen	do	3.56	4.97	4.73	
Dried in hydrogen	Ether	3.56	4.96	4.71	
Germ samples:					
Dried	do				41.79
Not dried	do				42.39
Wet germs	do				44.93
Dried	CS_2				41.85
Not dried	do				42.39
Wet germs	do				44.87

These results show very marked differences before and after drying. The greatest differences are found with the gluten meal, smaller ones with the gluten feed, still smaller with the bran, and very small variations with the germs. The physical condition of these products easily accounts for these results. The meal is in small balls and the first drying during the process of manufacture affects only the surface. The preparation of the sample for analysis, grinding so as to pass through a 1-mm sieve, exposes an immense amount of fresh surface to the direct action of the heat during the extra five to twenty-four hours' drying in the laboratory. The amount of fresh surface exposed with the other products is a great deal less, and therefore the differences in the extraction's results are less also. The extraction of samples of feed, grinding some before, and some after drying, shows differences of 0.1 to 0.3 per cent, the higher results being obtained on the samples that were dried in the coarse condition. The bran is in such a condition that a large surface is exposed in the original drying during manufacture and subsequent drying in the laboratory has very little further effect. The grinding of this product also exposes comparatively little new surface and the extraction figures, drying before and after grinding, do not differ materially. The gluten feed being a compound product of the other two, the results are between those of the two component parts. The germs are in the course of manufacture very thoroughly dried to below 3 per cent, and the subsequent drying of same in the laboratory gives only slightly lower figures. The fat in germs that were dried over sulphuric acid or not at all, will be from 2 to 4 per cent above that found in the same sample after drying. Corn oil in a very finely divided condition, as it exists in these feeds, will be easily partially oxidized and saponified. No appreciable differences were found by drying the feeds in air, vacuum, or hydrogen.

The change in the fat, due to this drying, affects the acidity of the feed and especially the acidity of the extracted fat. These factors are often determined and used as the basis of an opinion as to the character of the feed and of the raw material from which the feed was made. The acidity of the fat from gluten feeds, extraction having been made after drying, is from 20 to 40 per cent determined as oleic acid. The acidity of fat extracted before drying will be from 5 to 15 per cent.

The fat from dry germ oil cake will show as high as 60 per cent acidity. This can be explained and no doubt is due to the high heat and steaming of the finely ground germs before being pressed. Some of the figures obtained from one of the experiment stations some time ago agree very closely with results obtained by myself.

TABLE II.—*Determinations of fat and acidity in gluten feeds.*

[Experiment station work.]

Sample.	Fat in feed.	Acidity of feed.	Acidity of fat.	Oleic acid in fat.
	Per cent.	*cc N/10 alkali.*	*cc N/10 alkali.*	*Per cent.*
A	3.16	2.8	1.05	46.8
B	2.48	4.85	.55	39.1
C	2.60	4.80	.90	48.8
D	3.43	.75	.85	35.0
E	3.78	7.10	.65	24.3

The highest percentage of acid, found in sample E, is accompanied by the lowest acidity in the extracted fat. Sample C contains a higher percentage of acidity in the extracted fat than was originally found in the feed. If the acidity of the feed is exactly neutralized, the fat extracted without drying will be neutral, while the fat extracted after drying will again show acidity. No doubt a saponification takes place, which will account both for the lower percentage of fat found and for the higher acidity of the same. The determination of the acidity of the feed is also of no value as an indication of the quality of the feed. All cereals give acid reactions. If the gluten feed or meal has been made by the old alkali process, the presence of sulphuric acid can be easily determined. In the new process, the acidity is due to acid salts and proteids, but not to free acid. The acidity determination can not be made directly on the feeds, as color, proteids, and starch products interfere with the action of the alkali and the indicators. Washing out the acid is a very tedious operation, especially as large quantities must be used to get average figures. The following method I have found to give very satisfactory results:

Weigh out 100 grams, place in a large beaker or flask with 1,000 cc of water; stir or agitate every now and then for one hour. Pour the whole mass on a double filter paper. Reject the first 100 cc of the filtrate. Take 200 cc for the determination, figuring 10 grams of substance to 100 cc of the filtrate. Unless the acidity is very low, use normal caustic alkali for the titration; $\frac{1}{10}$ cc will give a quick color reaction with the indicators used. One can not titrate to a permanent shade, as the substances held in solution react both on the alkali and on the indicators. The error due to the strength of the titrating solution is much reduced by the large amount of substance titrated, 20 grams per test. The indicators used play an important rôle in the amount of acidity found. The average of a large number of tests gave the following results:

	cc normal alkali.
Phenolphthalein	11.1
Rosolic acid	6.7
Congo red	5.6
Litmus	5.5
Cochineal	4.0
Methyl orange	Neutral to alkaline

I prefer, and use, rosolic acid. Phenolphthalein gives very high results. Gluten feeds contain the proteids of maize and these react on indicators similarly to the proteids of wheat described by Osborne, who states[a] that the proteids of wheat are "neutral to litmus, alkaline to lacmoid," and that "it required 19 cc of decinormal alkali to neutralize 100 cc, using phenolphthalein."

Tests made to determine whether the fermentation of the feeds before drying had any effect on the finished products gave the following results:

TABLE III.—*Effect of fermentation before drying on acidity of gluten feeds.*

Condition of feed.	Acidity, measured by normal alkali, as indicated by—	
	Phenolphthalein.	Rosolic acid.
Before drying:	*cc.*	*cc.*
Unfermented	7.3	7.2
Fermented	28.3	27.1
After drying:		
Unfermented	12.3	7.4
Fermented	12.5	7.5

This proves conclusively that the fermentation of the feed before drying does not affect the finished product at all, and that the determination of the total acidity of these feeds is not a criterion as to the condition of the feeds before drying. The statements that a high acidity of gluten feeds shows that they were made from fermented products are not borne out by facts. All corn feeds show a higher acidity after drying than before. The drying of these feeds increases the solubility of the proteids, and changes the starch into gums and dextrins, also soluble.

TABLE IV.—*Effect of drying on acidity of gluten feeds.*

Feed and condition.	Extract as percentage of dry substance.	Acidity, measured by normal alkali, as indicated by—	
		Phenolphthalein.	Rosolic acid.
Gluten meal:	*Per cent.*	*cc.*	*cc.*
Before drying	1.2	0.4	0.4
After drying	2.1	.6	.5
Gluten feed:			
Before drying	1.2	6.0	5.8
After drying	6.2	11.7	6.0
Corn bran:			
Before drying	6.7	11.5	11.6
After drying	11.3	21.3	12.0

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[a] Am. Chem. Journal, July, 1900.

When the extract contains about 2 per cent of dry substance, the percentage of crude proteids will be about 4 per cent of the same. As the extract increases the proteids increase, and an extract containing 10 to 12 per cent of dry substance will show up to 35 per cent of proteids ($N \times 6\frac{1}{4}$). No definite ratio exists between the extract and its content of proteids. The results obtained in the determination of fat, acidity of fat, and acidity of feeds can not be used in direct comparison with results obtained on other cereal products. The quality of the feeds can not be determined from the acidity of the feed nor acidity of the extracted fat. All these results are materially affected by the treatment given the samples in the laboratory and in justice to the manufacturer, and so as not to mislead the consumer in comparing analyses of feeds the analyses should be made on the samples as received by the analyst, instead of modifying them to meet the requirements of any special or universal method of analysis.

REPORT ON FOOD ADULTERATION.

By W. D. Bigelow, *Referee.*

Since our meeting a year ago preliminary reports have been prepared giving methods for the examination of cereal products and tea and coffee. This leaves but one subject which has not already been covered by our methods, and the omission in that case has been due to the serious illness of the referee to whom the subject was assigned.

Considerable work has been done during the past year in putting to further test the methods which have been adopted provisionally for the examination of foods. The result has been the suggestion of certain changes which are relatively unimportant, and whose number is so great as to make it impracticable to discuss them in detail before the association. Owing to this fact, and also to the fact that there are very few here this morning who have given special attention to methods for the analysis of foods, or who are greatly interested in the detail of these methods, I shall pass over the suggestions of minor changes with the statement that they meet with the approval of myself and associates, and with the recommendation that they be incorporated in the provisional methods and printed in order that they may be available to the association.

So far our work has been conducted as individuals. A few years ago comparatively few of our members were interested in methods of this nature. Since then the work has been taken up in many laboratories that have always been represented in the association, and the chemists of food-control laboratories in various States and cities look to us to suggest methods for their use and cooperate with us in the preparation of those methods. I believe that we have made sufficient progress with these methods to make it practicable to carry on collaborative work between different laboratories in order to investigate and compare the methods we employ. From this time on, therefore, we can work with the methods for the examination of foods in very much the same way as we have with those for the examination of fertilizers, soils, and feeding stuffs.

It was only by individual effort that the methods could have been mapped out on the extensive scale which has been adopted, but systematic collaborative effort will be necessary to work out these methods in the detail that is desired. As previously stated, I do not think it would be wise to discuss before the whole association the changes which we recommend this year in the provisional methods. At the same time, following the collaborative work which we plan for the coming year, I believe that provision should be made at future meetings of the association for the detailed discussion of the methods for the examination of foods, and that hereafter the conservative attitude which the association has adopted with regard to other methods of analysis be extended also to food methods.

In closing I wish to express my appreciation for the able and earnest manner in which my associates have conducted their work.

The **President**. You have heard the referee's report on foods. Is there any discussion of the subject?

Mr. **Bigelow**. Perhaps it is not related to this subject, but I have a suggestion to offer which I was requested to make by Dr. Wiley. He asks that as many members of the association as can find it convenient take dinner to-morrow evening at Cabin John Bridge. He did not say that we were to be there as his guests, but he would be glad to have as many go as possible. The accommodations are good, and it is a nice ride, which I am sure all of you would enjoy. All who can find it convenient to start from the Ebbitt House soon after 5 o'clock we shall be glad to have go, and if they will hand me their names, I will try to make some arrangements.

The **President**. If there is no other business to come before the meeting we will listen to the report of the referee on the determination of nitrogen.

REPORT ON THE DETERMINATION OF NITROGEN.

By F. W. Morse, *Referee.*

The referee, in accordance with the vote of the association last year, undertook the comparison of methods for determining the availability of organic nitrogen. Two methods were recommended by the previous referee for trial, namely, the neutral permanganate method, as modified by Mr. Street, and the alkaline permanganate method, as modified by Mr. Jones. The former has been adopted by this association as a provisional method, while the latter has not been regarded with much favor by the majority of analysts who have tried it.

Since nearly all the principal nitrogenous fertilizer materials have been worked upon in the past by the different methods, the referee considered it the best policy this year to confine the work to two typical materials combined with superphosphate in approximately the proportion which might occur in a mixed fertilizer, in order to determine how closely results by different analysts would agree, and to make plainer any defect in the manipulation of the method.

The materials selected were dried blood and cotton-seed meal, because both are known to be highly available to plants, while the alkaline method has always failed to indicate the availability of cotton-seed meal, owing apparently to its carbonaceous matter. The blood was a part of a bag of blood meal for poultry and calves, and contained 13.1 per cent of nitrogen. The cotton-seed meal was a standard brand and contained 7.49 per cent of nitrogen.

Four lots of material were prepared containing approximately 3.5 to 4 per cent of nitrogen. The mixtures all contained superphosphate while the nitrogenous material was arranged as follows: No. 1, blood; No. 2, cotton-seed meal; No. 3, two-thirds nitrogen from blood, one-third nitrogen from cotton-seed meal; No. 4, one-third nitrogen from blood, two-thirds nitrogen from cotton-seed meal.

All the materials were sifted through the standard sieve, with circular holes 1 millimeter in diameter, and then each lot was thoroughly mixed. The blood was apparently coarse because a large part of it was crystalline rather than fibrous. Two of the analysts reground the samples containing it.

Twelve lots of samples were sent to as many different laboratories. Accompanying the samples were sent the following instructions:

DIRECTIONS FOR WORK ON NITROGEN, 1902.

Please determine total nitrogen in each sample by either of the official methods. Last year the referee called attention to the fact that prolonged digestion with the sulphuric acid in the Kjeldahl method raised the amount of nitrogen. Please note the length of time used in each digestion throughout the comparison. Please determine available nitrogen by the neutral permanganate method adopted provisionally at the last convention, and by Jones's alkaline permanganate method, which has so far been regarded unfavorably. The latter method used in accordance with Mr. Jones's practice offers some advantages over the former in the matter of time. The methods are described as follows:

The neutral permanganate method.—Into a 400-cc Griffin beaker, low form, weigh an amount of sample containing, approximately, 0.075 gram of nitrogen (samples containing material that has been treated with acid should be washed on a 9-cm S. S. No. 595 filter to 200 cc and transferred, filter and all, to beaker); digest this with 125 cc of permanganate of potash solution (16 grams of pure $KMnO_4$ to 1,000 cc of water) in a steam or hot-water bath for thirty minutes. Have the beaker let down well into the steam or hot water and keep closed with a cover glass, stirring twice at intervals of ten minutes with a glass rod. At the expiration of the time remove from bath, add 100 cc of cold water and filter through a heavy 15-cm folded filter. Wash with cold water, small quantities at a time, till total filtrate amounts to 400 cc. Dry and determine nitrogen in residue by Kjeldahl method.

The alkaline permanganate method.—Weigh out an amount of sample containing, approximately, 0.045 gram of nitrogen, and transfer to a 600-cc distillation flask. After connecting with condenser to which the receiver containing standard acid has been attached, digest with 100 cc of alkaline permanganate solution (16 grams of potassium permanganate and 150 grams of sodium hydrate dissolved in 1,000 cc of water) for 30 minutes below the boiling point. Then boil slowly until the distillation is completed.

Since the samples all contain superphosphates, it will be necessary to wash them as directed for the neutral method. This is not necessary for the alkaline method, as it is intended to be used. Comparisons of washed and unwashed charges will be appreciated however.

The troublesome features of these methods seem to be filtration in the neutral and distillation in the alkaline method. Suggestions for hastening these steps will be of interest.

RESULTS OF ANALYSES.

The methods were copied from the report of last year.

Returns were received from nine laboratories and included the work of twelve analysts. The following tables give their results in full:

TABLE I.—*Total nitrogen.*

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	
T. C. Trescot, Washington, D. C.	3.78	3.30	3.82	3.73	Digested 2 hours.
	3.75	3.34	3.85	3.73	
	3.98	3.59	3.95	3.87	Digested 3 hours.
	4.09	3.60	3.95	3.85	
Department of foods and feeding, Hatch Experiment Station, Massachusetts	4.16	3.58	4.01	3.84	
	4.16	....	3.99	3.84	Digested 3 hours, Kjeldahl method.
	4.15	3.56	3.97	3.81	
	4.17	3.64	4.07	3.96	Digested 3 hours, Kjeldahl with sulphate of potash.
	4.14	....	4.06	3.92	
	4.14	3.63	4.03	3.92	
F. B. Carpenter, Virginia	4.18	3.59	4.06	3.97	
S. H. Sheib, Virginia	4.19	3.55	4.07	3.94	

TABLE I.—*Total nitrogen*—Continued.

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	
L. A. Hill, New Hampshire	3.90	3.54	3.75	3.67	Digested 3 to 4 hours.
	3.98	....	....	3.78	
	4.00	3.54	3.89	3.73	
W. W. Braman, New Hampshire.	3.98	3.45	3.88	3.84	
	4.03	3.48	3.94	3.86	
A. L. Sullivan, New Hampshire	4.05	3.33	3.92	3.64	Gas pressure lower than normal.
	4.06	3.33	3.89	3.72	
J. Bernard Robb, Maryland	4.03	3.50	3.95	3.77	Digested 1 hour.
	4.05	3.47	3.93	3.79	
	4.08	3.51	3.96	3.82	
C. H. Jones, Vermont	4.32	3.69	4.04	4.04	Digested 2½ to 3 hours.
	4.35	3.69	4.04	3.97	
	4.32	3.76	4.11	3.97	
F. W. Woll and Geo. A. Olson, Wisconsin.	4.16	3.58	4.01	3.93	Digested from 1½ to 5 hours.
	4.19	3.58	4.01	3.98	
	4.17	3.60	4.08	3.95	
	4.14	3.57	4.07	3.95	
	....	....	4.09	....	
W. H. Scherffins and A. M. Peter, Kentucky.	4.08	3.49	3.97	3.79	Digested 3 hours.
	4.07	3.53	3.88	3.86	
	4.13	3.54	3.95	3.86	Digested 5 hours.
	4.12	3.54	3.94	3.87	
W. R. Perkins, Mississippi	4.02	3.48	3.89	3.90	

TABLE II.—*Insoluble nitrogen—Neutral permanganate method.*

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	
T. C. Trescot, Washington, D. C.	0.68	0.37	0.94	0.72	Washed.
	.80	.25	.86	.83	
	1.90	.46	.91	.60	Not washed.
	1.78	.25	.72	.81	
Department of foods and feeding, Hatch Experiment Station, Massachusetts.	1.85	.40	1.22	.93	
	1.78	.39	1.15	.83	
S. H. Sheib, Virginia	.65	.30	.56	.61	
L. A. Hill, New Hampshire	.42	.31	.70	.84	Not washed.
	.46	.56	.78	.80	
	.53	.23	.49	.52	
	.61	.31	.50	.40	
W. W. Braman, New Hampshire.	.92	.56	.87	.77	
	.40	.36	.56	.52	
A. L. Sullivan, New Hampshire	1.01	.38	.67	.50	
	1.28	.41	.67	.52	
C. H. Jones, Vermont	1.29	.35	1.06	.87	
F. W. Woll and Geo. A. Olson, Wisconsin.	.34	1.17	1.04	1.52	
	.36	1.12	.99	1.55	
W. H. Scherffins and A. M. Peter, Kentucky.	.39	1.20	.97	1.51	
	1.26	.45	1.48	.97	
	1.47	.49	1.33	.76	
	1.26	.36	1.53	.83	
	1.34	.32	1.05	.91	
W. R. Perkins, Mississippi	.59	.41	.70	.71	
	.59	.41	.68	.64	

TABLE III.—*Soluble nitrogen—Alkaline permanganate method.*

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	
T. C. Trescot, Washington, D. C.	2.46	1.57	1.99	1.68	
	2.32	1.51	2.10	1.82	
Department of foods and feeding, Hatch Experiment Station, Massachusetts.	2.38	1.30	1.83	1.55	
	....	....	....	1.53	
	2.35	1.30	1.82	1.51	
L. A. Hill, New Hampshire	2.73	1.82	2.31	1.82	Distilled very low.
	2.80	1.96	2.38	2.13	
W. W. Braman, New Hampshire.	2.99	2.00	2.71	2.57	Do.
	2.98	1.93	2.80	2.49	
A. L. Sullivan, New Hampshire	2.90	2.44	2.41	2.26	Do.
	2.97	2.27	2.57	2.54	
J. Bernard Robb, Maryland....	2.83	1.85	2.40	2.27	
	2.88	1.84	2.46	2.17	
	2.75	1.78	2.31	2.19	
C. H. Jones, Vermont	2.67	1.50	2.16	1.86	
	2.64	1.53	2.09	1.90	
F. W. Woll and Geo. A. Olson, Wisconsin.	2.22	1.28	1.81	1.77	
	2.33	1.18	1.84	1.78	
	2.27	1.40	1.96	1.75	
	2.17	1.33	1.79	1.70	
W. H. Scherffius and A. M. Peter, Kentucky.	1.64	1.25	1.47	1.37	
	1.79	1.25	1.41	1.36	
	1.83	1.26	1.34	1.48	
W. R. Perkins, Mississippi	2.81	1.56	2.31	2.05	
	2.79	1.62	2.47	2.18	
	2.75	1.54	2.34	2.07	

TABLE IV.—*Availability of nitrogen in neutral permanganate.*

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	
T.C.Trescot, Washington, D. C.	81.63	91.36	77.21	80.05	Washed until neutral.
	54.34	90.25	79.49	81.86	Unwashed.
Department of foods and feeding, Hatch Experiment Station, Massachusetts.	55.45	89.00	69.64	76.05	
	57.21	89.15	71.24	78.53	
S. H. Sheib, Virginia...........	84.49	93.52	86.24	81.97	
L. A. Hill, New Hampshire	88.97	87.57	80.62	77.68	Unwashed samples.
	85.75	92.38	86.85	87.94	Washed samples.
W. W. Braman, New Hampshire.	83.50	87,57	81.84	83.36	
A. L. Sullivan, New Hampshire	71.85	88.28	82.82	81.70	
C. H. Jones, Maryland	70.20	90.57	73.89	79.45	
F. W. Woll and Geo. A. Olson, Wisconsin.	91.36	67.31	75.30	61.26	
W. R. Perkins, Mississippi.....	85.32	88.31	82.26	82.69	

TABLE V.—*Availability of nitrogen in alkaline permanganate.*

Analyst.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
T. C. Trescot, Washington	59.30	42.89	51.64	45.53
Department of foods and feeding, Hatch Experiment Station, Massachusetts.	57.26 .... 56.68	56.22 .... 36.04	45.45 .... 45.30	40.00 39.47 39.00
L. A. Hill, New Hampshire	69.17	53.39	61.53	52.95
W. W. Braman, New Hampshire.	74.50	56.64	70.33	65.71
A. L. Sullivan, New Hampshire	72.34	70.57	63.84	65.21
J. Bernard Robb, Maryland	69.63	52.00	60.91	58.15
C. H. Jones, Vermont	61.31	40.88	52.34	47.12
F. W. Woll and Geo. A. Olson, Wisconsin.	54.19	36.31	45.43	44.30
W. R. Perkins, Mississippi	69.23	45.21	61.01	53.84

COMMENTS OF ANALYSTS.

Department of Foods and Feeding, Hatch Experiment Station.—Mechanical conditions were not uniform; a considerable portion of each sample was coarse. The substances should be reduced so as to pass a sieve of No. 2 perforated sheet metal (400 perforations to the square inch). This is especially true of mixtures containing highly nitrogenous foods; otherwise sampling for nitrogen determinations or availability, where only a small amount is employed, is very unsatisfactory, and concordant results are practically impossible except by chance.

In the determination of total nitrogen, by the plain Kjeldahl method, the reagents used in the digestion were one-half gram resublimated metallic mercury (B. and A.), 25 cc chemically pure sulphuric acid, specific gravity 1.84 (B. and A.); and, in the distillation, 200 cc distilled water, 25 cc potassium sulphid solution, 1–25 (Merck), 100 cc sodium hydrate solution, 1–2 ("Red heart"), one-half gram granulated zinc (30 mesh, B. and A.)

In the determination of total nitrogen by the plain Kjeldahl method plus sulphate, the reagents used were the same as in the plain Kjeldahl with the addition of 10–12 grams of crystallized potassium sulphate (B. and A.), to facilitate the digestion and to insure more perfect oxidation.

The results were as follows:

(1) More perfect oxidation; this is noticeable in every case where cotton-seed meal is present; the average increase in nitrogen for the samples is 0.056 per cent, 3.943 per cent, and 3.887 per cent.

(2) More rapid oxidation; the solutions cleared—i. e., bulk of organic matter was destroyed in twenty-one minutes (average) as against forty-one minutes in the plain Kjeldahl method.

(3) There is less reaction upon the addition of water in preparing for the distillation than in the case of the plain Kjeldahl method.

The mechanical condition prevented the obtaining of closely agreeing results in both methods.

The neutral permanganate solution was prepared from pure crystallized reagent (Merck). The plain Kjeldahl method plus sulphate was used for the determination of the nitrogen in the insoluble residue. Unevenness of the samples and variation in action of the solvents probably account for the poor parallels. The objectionable features in the manipulation of this method are chances of error from repeated transferring, etc., length of time required, and difficult filtration.

The alkaline permanganate solution was prepared from pure crystallized potassium permanganate (Merck) and stick sodium hydrate, purified by alcohol (Merck). The results are apparently low. This can be accounted for, partly at least, by errors in the method as sent out:

(1) A weak, incorrectly prepared solution. Sixteen grams of powdered crystallized potassium permanganate and 150 grams of sodium hydrate should be dissolved in water, and after cooling made up to 1,000 cc.

(2) Substances too coarse to receive the maximum effect of the solution.

(3) Digestion too short. The heating should be maintained for one hour without distilling over, but not necessarily below ebullition, and the flask should be shaken occasionally to wash down adhering particles. The distillation should be conducted as usual, simply using care not to break the flask. The objectionable feature in the manipulation is the adhering of the material to the sides of the flask and the smallness of the solution with which to wash it down. Little or no frothing occurs after proper digestion.[a]

C. H. Jones, Vermont.—The filtrate after the digestion with neutral permanganate solution was colorless in each case.

With the alkaline permanganate method I am satisfied that the preliminary digestion of thirty minutes or one hour is necessary, not so much that the results may be slightly increased, but that danger from frothing is lessened if not entirely obviated when the distillation begins. It should be noted that it is quite necessary to rotate the flask occasionally during the digestion and distillation if the material shows a tendency to creep up the sides of the flask.

I reground samples Nos. 1, 3, and 4 before making any tests, as the samples containing blood seemed to me too coarse to enable one to get concordant results with the Kjeldahl method.

J. B. Robb, Maryland.—The total nitrogen was determined by the Gunning method and the samples were digested for one hour.

In the alkaline permanganate method the flasks were shaken several times during the process of distillation to prevent the material from adhering to the sides of the flask.

In my opinion the neutral permanganate method is very unsatisfactory, as the results obtained by it are not at all concordant.

F. W. Woll, Wisconsin.—No. 597, S. & S. filters 9 cm were used.

Sample No. 1, blood, washed clear.

Sample No. 2, cotton-seed meal, washed turbid, which necessitated refiltering through double paper (No. 597, S. & S.).

Sample No. 3, washed clear.

Sample No. 4, washed turbid, treated the same as sample No. 2.

Jena 800-cc round-bottom flasks were used for distillation.

A. M. Peter, Kentucky.—It being apparent that all the permanganate was reduced before the digestion was completed, in the alkaline method, an additional quantity of 1.6 grams permanganate dissolved in 100 cc water were added to each of the flasks in set 3 and the distillation repeated, the following additional quantities of nitrogen being obtained: Sample No. 1, 1.25 per cent; No. 2, 1.30 per cent; No. 3, 1.54 per cent; No. 4, 1.40 per cent. At the close of the distillation the liquid in the flasks was still green from excess of manganate.

Another portion of 1.6 grams permanganate and 100 cc water was added to each flask and the distillation was repeated, with the following results: Additional nitrogen obtained from sample No. 1, 0.40 per cent; No. 2, 0.20 per cent; No. 3, 0.20 per cent; No. 4, 0.13 per cent.

The total available nitrogen obtained in these three distillations was as follows:

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[a] See the Fourteenth Report of the Vermont Experiment Station, p. 219.

Sample No. 1, 3.22 per cent; No. 2, 2.76 per cent; No. 3, 3.08 per cent; No. 4, 3.01 per cent.

Another set of determinations was made, following the referee's directions in every respect, except that 200 cc of alkaline permanganate were used instead of 100 cc. The results were that the following percentages of available nitrogen were obtained: Sample No. 1, 2.24; No. 2, 1.90; No. 3, 2.17; No. 4, 2.10.

Still another series was made, following the referee's directions in every particular, except that the distillation was pushed until the residues in the flasks were dry at the bottom. In this set some parts of the residue may have been overheated, as the flasks were over the direct flame. The results were the following percentages of available nitrogen: Sample No. 1, 2.58; No. 2, 2.16; No. 3, 2.93; No. 4, 1.50.

It appears that there is not enough permanganate in 100 cc of the alkaline solution as prescribed to complete the decomposition. I do not see that either method is giving us available nitrogen, and the alkaline permanganate, as carried out, seems to be especially faulty.

DISCUSSION OF RESULTS.

Without discarding any figures on total nitrogen, the differences between maximum and minimum results are, respectively, 0.60 in No. 1, 0.46 in No. 2, 0.36 in No. 3, and 0.40 in No. 4. These discrepancies are large for total nitrogen.

The point mentioned by the referee last year with regard to the time of digestion is brought out here, namely, that the digestion should be prolonged in order to insure the recovery of all the nitrogen.

Setting aside the highest and lowest results we have determinations ranging within less than 0.3 per cent, which is as exact a result as has been obtained in the past.

When the results on the availability of the nitrogen are considered, it is seen that duplicate analyses by any one analyst agree well, taking each method by itself.

Both methods have been evolved from the method of determining albuminoid ammonia in water, as is shown by the first method proposed in the reports of the association.[a] They differ radically in principle, the neutral permanganate being used merely to dissolve the nitrogen, while the alkaline method dissolves it and also transforms it into ammonia.

The critical features of the former method are time and temperature, as in determining citrate soluble phosphoric acid. The latter method possesses the same weakness possessed by Wanklyn's method for albuminoid ammonia—that in the case of vegetable matter it will recover only about one-half of the total nitrogen, and its critical features are the amount of permanganate which will be affected by cellulose and the amount of distillate which is collected.

As is mentioned by Mr. Peter, neither method seems to give the available nitrogen; yet I can think of nothing better. We have in fertilizers about all forms of vegetable and animal proteids, both raw and cooked. Obviously none of the usual solvents for proteids can be employed in this work. We must therefore attempt to use a substitute.

The neutral permanganate method has compared most favorably with vegetation tests in its relative effects on the various nitrogenous fertilizers. On the other hand, the alkaline method is a convenient one for determining whether a fertilizer is easily transformed to ammonia or not.

This method needs to be modified somewhat. If more time had been available, the referee would have tried increasing the amount of solution used in order to have a greater volume for distillation and the regulation of the amount of distillate collected. Reasoning from the practice with albuminoid ammonia in water analysis, this latter point would be especially important. Another fact which bears on this point is that in residues after distillation, when washed as in the neutral method and the insoluble nitrogen determined, the nitrogen was found to have been almost totally dissolved, even in a sample of ground leather.

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[a] U. S. Dept. Agr., Division of Chemistry Bul. 47.

With regard to the neutral method I think that the use of a Witt plate or Hirsh funnel is, as a rule, advantageous in reducing the time and in enabling one to use a smaller filter paper.

I do not think it necessary to make a change in the method of determining total nitrogen, although the weight of evidence is in favor of prolonging digestion for three or more hours. The increase in nitrogen is so slight that it would be negligible in some work, while important in others. The good sense of our station chemists will result in their familiarizing themselves with the work of the association on such a point, after which the character of the work will govern the mode of execution.

I believe that the alkaline permanganate method is worthy of further attention, and that each method should be reduced to a form which will give more closely concordant results.

My only recommendation as a result of this year's work is that the subject be continued by the next referee, with the view of getting more concordant results with each method.

The **President**. Is there any discussion on this subject? There was a time when this association discussed the papers submitted by referees.

Mr. **Cavanaugh**. In determining nitrogen in the Cornell University agricultural experiment station laboratory, both mercuric oxid and potassium sulphate are used. The order of adding the potassium sulphate and sodium hydroxid, as given in the present official method, is reversed. The sodium hydroxid is added first, and the precipitated mercurammonium compound is then decomposed by the potassium sulphate. That complete decomposition takes place has been repeatedly demonstrated by many trials, comparing this modification with the official methods. The advantage gained is in adding the potassium sulphid to an alkaline solution, thereby avoiding an evolution of hydrogen sulphid. The time of digestion is shorter when both mercuric oxid, or mercury, and potassium sulphate are used than with either alone. Distillation with steam has been found very efficient, especially in soil work. No bumping ever occurs even when there is a large amount of precipitate in the distilling flask.

The **President**. I will now call for the report on sugar.

REPORT ON SUGAR.

By G. L. Spencer, *Referee.*

The associate referees, Messrs. L. S. Munson and L. M. Tolman, have conducted the experimental work on chemical and optical methods in sugar analysis. No report is presented by Mr. D. L. Davoll, who was appointed to investigate special analytical methods used in the sugar industry.

Outline of Work Done.

I shall present a brief outline of the investigations made, and from these and the results of my own experience in those analytical methods of special importance in the sugar industry, recommendations will be offered upon optical and chemical methods and upon that part of the work which was allotted to Mr. Davoll.

Mr. Munson's investigations were made with a view to the simplification of the methods for determining reducing sugars and increasing the accuracy of such work. He reviewed the work of many chemists, and conducted analyses for the verification of their results. He made experiments to determine whether the copper in the cuprous oxid obtained in the reduction method can be accurately ascertained from the weight of the suboxid. His results fully demonstrate the accuracy of the method in which the suboxid is directly weighed. Mr. Munson also conducted investigations along the line of devising a single copper solution which could be used with all reducing sugars with a uniform length of time and method of heating. This work has led to no definite conclusions, but the outlook is sufficiently promising to warrant continuing these investigations another year. He also investigated various conditions which modify the reduction. The results of Mr. Munson's investigations will be given in a paper which he will read at the proper time.

The work of Mr. Tolman was in the study of optical methods for the determination of sucrose. Mr. Tolman made experiments to show the effect of varying quantities of acid in the inversion upon given quantities of sugar, and made a study of the effect of these changes upon the factor used in the calculations. He also made a study of inversion at ordinary laboratory temperatures, with a view to simplifying the optical method usually termed Clerget's method.

Mr. Tolman investigated the use of hydrochloric acid in the proportions used in the German official method, in the inversion of sucrose at ordinary temperatures and obtained very satisfactory results. His investigations and the very large amount of work that has been done abroad in the verification of the German official method render it advisable that we adopt it as the official method of this association.

Mr. Tolman also investigated the use of citric acid in the inversion of sucrose, and demonstrated that in the absence of sodium acetate it will give very accurate results. Its advantage lies in not affecting the rotatory power of levulose or decomposing it. This method of inversion is of value in the examination of honeys. Mr. Tolman has prepared a paper for the association, giving his investigations in full.

In the matter of special analytical methods used in the sugar industry, those which should claim the attention of this association are methods relating to the analysis of sugar beets, with a view to fixing their purchase price, and the methods for the analysis of raw sugars.

In our opinion only direct methods of beet analysis need be considered. These methods are divided into two classes, viz, alcoholic and aqueous methods. The alcoholic methods are usually considered the more scientific and are largely employed in sugar-house control. The Germans usually prefer the alcohol methods. The aqueous methods, devised by Pellet, are used almost exclusively in France and Belgium and to a large extent in Germany and in the United States. Owing to the expense incurred in the alcoholic methods for the solvent, and also to the time involved, the water methods and the indirect method are used almost if not quite exclusively in this country in testing beets to fix the purchase price.

The question of the examination of raw sugars has been very thoroughly considered by the Treasury commission and by the committee of the international congress of applied chemistry and we shall base our recommendations largely upon their work.

RECOMMENDATIONS.

The referee and his associates offer the following recommendations for changes in and additions to the official methods and for future work:

(1) To the heading "Determination of water," page 27,[a] add "and density;" after (*a*) insert "water," and after (*b*) change to read "Areometric methods for density and indirect estimation of water."

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[a] References are to Bul. No. 46, revised, Div. of Chem., U. S. Dept. of Agr.

(2) Page 28: Insert the following to precede the table:

This method is accurate with liquids containing only soluble carbohydrates. With sirups, molasses, and liquids containing mineral matter, the results are only roughly approximate.

(3) Page 38: Insert the following method for ascertaining the amount of copper reduced, as a provisional method:

(*f*) METHOD BY DIRECT WEIGHING OF THE CUPROUS OXID: Prepare a Gooch crucible for the filtration by loading it with a felt of asbestos one-fourth inch thick. First thoroughly wash the asbestos with water to remove small particles, then follow successively 10 cc alcohol and 10 cc of ether, and dry the crucible and contents thirty minutes in a water oven at 100° C

Collect the precipitated suboxid of copper in the felt as usual, thoroughly wash it with hot water, then with 10 cc of alcohol, and finally with 10 cc of ether. Dry the precipitate thirty minutes in a water oven at 100° C; cool and weigh it. The weight of cuprous oxid multiplied by 0.888 gives the weight of metallic copper.

(4) Page 39, "Optical methods for the determination of sucrose," omit Clerget's method (page 39) and Creydts' method (page 40), and substitute for them the German official methods, as follows:

Determination of sucrose and raffinose.—This method is not applicable in the presence of optically active bodies other than sucrose and raffinose. Percentages of raffinose less than 0.33 can not be determined with certainty by this method.

Dissolve the normal weight of the material in water, clarify as usual, and dilute to 100 cc. Filter and polarize the filtrate at 20° C. Record the polariscope reading as the direct reading or polarization before inversion.

Dissolve 13.024 grams of the substance in 75 cc of water in a 100-cc flask, and add little by little, while rotating the flask, 5 cc of hydrochloric acid containing 38.8 per cent of the acid; and heat the contents of the flask, after mixing, on a water bath heated to 70° C. The temperature of the solution in the flask should reach 67° to 70° in two and one-half to three minutes. Maintain a temperature of as nearly 69° as possible during seven to seven and one-half minutes, making a total time of heating of ten minutes. Remove the flask and cool the contents rapidly to 20° C., and dilute the solution to 100 cc. Polarize this solution in a tube provided with a lateral branch and a water jacket passing a current of water around the tube to maintain a temperature of 20° C.

Multiply the invert reading, made at 20° C. by 2. If a preliminary calculation using the formula,

0.7538X (algebraic difference of the direct and invert readings) = per cent sucrose,

gives a percentage which is more than 1 per cent higher than the direct reading, raffinose is probably present, and the following formulæ by Creydt should be used:

P—the direct reading.
I—the invert reading.
S—the percentage of sucrose.
R—the percentage of anhydrous raffinose.

$$S=\frac{0.5188P-I}{0.8454};\ R=\frac{P-S}{1.85}.$$

Determination of sucrose in the absence of raffinose.—The inversion and observation should be made as described above and the following formula used:

When the polarizations are made at 20° C.:

$$S=\frac{100\ (P-I)}{142.66-\frac{20^\circ}{2}}=0.7538X\ (P-I).$$

Should the temperature (t) vary from 20°, which is permissible within narrow limits, in the absence of raffinose use the following formula:

$$S=\frac{100\ (P-I)}{142.66-\frac{t}{2}}.$$

It is recommended that it also be permissible to conduct the inversion as follows:

Prepare the solutions as described above, using 50 cc of water instead of 75 cc and employing the proportion of acid specified, and set the flask aside during a period of twenty-four hours at a temperature of not below 20° C.; or if the temperature be above 25° set aside ten hours.

(5) For the direct analysis of sugar beets we recommend that the following be inserted as provisional methods:

(*a*) *Scheibler's alcoholic method.*—In the direct analysis of the beet with the Soxhlet-Sickel apparatus by Scheibler's method, proceed as follows for the extraction of the sucrose:

Place a plug of absorbent cotton in the bottom of the tube, then introduce 26.048 grams of the pulped beet, or 2×16.29 grams, according to the polariscope in use, pressing the pulp lightly with a rod. Very small fragments of the beet may be used instead of pulp. Connect the extractor with the reflux condenser. Place 75 cc of 95 per cent alcohol in the flask and connect with the extractor; heat the flask in the water bath and continue the extraction from half an hour to two hours or more, according to the state of division of the sample. Use somewhat weaker alcohol if only 16.29 grams of pulp be taken. Cool and remove the flask, substitute a second containing 75 cc of 75 to 80 per cent alcohol, and continue the extraction to ascertain whether the first extraction were complete.

Fill the first flask to the 100-cc mark, after treating the sample with two or three drops of subacetate of lead solution. Mix the contents of the flask, filter, and polarize. Having extracted the normal weight of pulp, the polariscopic reading is the per cent of sucrose in the sample.

The extract in the second flask should also be polarized as a check upon the extraction.

Great care is essential in the polarization of alcoholic solutions. The least quantity of subacetate of lead that will clarify the solution should be used. The solution must be protected from evaporation during the filtration by a cover glass. Avoid irregularities in the temperature of the solution in the observation tube, due to the warmth of the hands; since the density of the solution in different parts of the tube will vary under such conditions, striæ will form, rendering an accurate reading impossible.

The Scheibler method, as above described, differs from the original only in a few minor details, especially in the arrangement of the extraction apparatus. The Soxhlet extraction apparatus is much more effective than Scheibler's original instrument.

(*b*) *Pellet's aqueous method—Hot digestion.*—Any good rasp may be used in the preparation of the pulp for this method. The special flasks with enlarged necks are convenient for use in this method. Transfer 26.048 grams of the pulp to the flask, using a little water to wash the weighing capsule and funnel; or, for the Laurent, employ 32.58 grams of pulp, i. e., 2 × normal weight. The flasks are graduated to contain 201.35 cc for the Schmidt and Haensch and 201.7 cc for the Laurent polariscopes, in order to compensate for the volume of the marc and the lead precipitate. Add 5 to 10 cc subacetate of lead solution of 54.3° Brix for the clarification. Approximately 6 to 7 cc are required per 26 grams of beet pulp. This reagent should be run into the flask in advance of the beet pulp. Add a few drops of ether to beat down the foam, then sufficient water to increase the volume of the solution to about 190 cc. Heat to 80° C. in a water bath and maintain this temperature about thirty minutes, occasionally giving the flask a circular movement to facilitate the escape of the air from the pulp. Increase the volume of the solution from time to time during the heating, so that when the operation is completed only a few drops of water will be required to complete the volume of the solution to the mark. After approximately thirty minutes' heating, cool the flask and contents and add strong acetic acid to the solution to acidity, dilute to the graduation, mix, and filter. The state of division of the pulp will govern the time of heating. In polarizing the filtrate, use a 400-mm. observation tube, thus directly obtaining the per cent sucrose in the beet with the Schmidt and Haensch polariscope, or double this percentage if the Laurent instrument be used.

(6) For the analysis of sugars we recommend that the methods selected by the international committee for unifying methods of sugar analysis be adopted as provisional methods of this association. These methods are as follows:

1. In general, all sugar tests shall be made at 20° C.
2. The graduation of the saccharimeter shall be made at 20° C. Twenty-six grams of pure sugar, dissolved in water, and the volume made up to 100 metric cc, or during the period of transition 26.048 grams of pure sugar in 100 Mohr cc, all weighings to be made in air with brass weights, the completion of the volume and the polarizations to be made at 20° on an instrument graduated at 20° should give an indication of 100 on the scale of the saccharimeter. For countries where temperatures are usually higher than 20° C., it is permissible that saccharimeters be graduated at 30°, or any other suitable temperature, under the conditions specified above, provid-

ing that the analysis of the sugar be made at the same temperature—that is, that the volume be completed and the polarizations made at the temperature specified.

3. Preparation of pure sugar: Purest commercial sugar is to be further purified in the following manner: A hot saturated aqueous solution is prepared and the sugar precipitated with absolute ethyl alcohol; the sugar is carefully spun in a small centrifugal machine and washed in the latter with absolute alcohol. The sugar thus obtained is redissolved in water, the saturated solution again precipitated with alcohol and washed as above. The product of the second crop of crystals is dried between blotting paper and preserved in glass vessels for use. The moisture still contained in the sugar is determined and taken into account when weighing the sugar which is to be used.

The committee further decided that central stations shall be designated in each country which are to be charged with the preparation and distribution of chemically pure sugar. Wherever this arrangement is not feasible, quartz plates, the values of which have been determined by means of chemically pure sugar, shall serve for the control of the saccharimeters. The committee further decided that the above control of quartz plates by means of chemically pure sugar should, as a rule, apply only to the central stations which are to test the correctness of saccharimeters; for those who execute commercial analyses, the repeated control of the instruments is to be accomplished, now as before, by quartz plates.

4. In effecting the polarization of substances containing sugar, half-shade instruments, or triple field, only are to be employed.

5. During the observation the apparatus must be in a fixed position and so far removed from the source of light that the polarizing nicol is not warmed.

6. Sources of light may be gas, triple burner with metallic cylinder, lens and reflector; gas lamps with Auer (Welsbach) burner; electric lamp; petroleum duplex lamp; sodium light. Several readings are to be made and the mean thereof taken, but any one reading must not be neglected.

7. In making a polarization, the whole normal weight for 100 cc is to be used, or a multiple thereof for any corresponding volume.

8. As clarifying and decolorizing reagents there may be used: (*a*) subacetate of lead (3 parts by weight of acetate of lead, 1 part by weight of oxid of lead, 10 parts by weight of water); (*b*) alumina cream; (*c*) concentrated solution of alum. Boneblack and decolorizing agents are to be excluded.

9. After bringing the solution exactly to the mark, at the proper temperature, and after wiping out the neck of the flask with filter paper, all of the well-shaken clarified sugar solution is poured upon a dry rapidly acting filter. The first portions of the filtrate are to be rejected and the rest, which must be perfectly clear, used for polarization.

(7) For future investigations we recommend a continuation of the studies along the lines of those of this year.

The PRESIDENT. Are there any papers on this subject for presentation?

Mr. MUNSON. I have a paper on sugar to submit to the association.

METHODS FOR DETERMINING REDUCING SUGARS.

By L. S. MUNSON.

METHODS IN USE.

The methods now used by this association for the determination of reducing sugars are: (1) an approximate volumetric method for rapid work; (2) Soxhlet's volumetric method for more exact work; (3) Herzfeld's method for analysis of mixtures containing less than 1 per cent of reducing sugar; (4) Meissl and Hiller's method for analysis of mixtures containing from 1 per cent to 100 per cent of reducing sugars; (5) Allihn's method for the determination of dextrose; and (6) Soxhlet's method for the determination of milk sugar. No method has ever been accepted by the association for the determination of maltose.

The methods for invert sugar and dextrose were adopted at the ninth annual meeting of the association, and have been used to the present time without any modification. Soxhlet's method for milk sugar was adopted at a later date, and is, without modification, the method devised by Soxhlet in 1880. The volumetric methods are but little used, except in sugarhouse control, where rapidity is essential. The gravimetric methods for both invert sugar and dextrose are so extensively used in agricultural chemical analysis that it was thought advisable to spend the time of the associate referee in work upon these methods. In outlining this work with the referee, it was decided that an attempt should be made to devise a common method for all the reducing sugars, and to construct tables giving values of the reduced copper in terms of the various sugars. Such a method would greatly simplify the reducing sugar proposition by doing away with the necessity of having different solutions for each of the reducing sugars, and in case of mixtures it would afford a more satisfactory basis for calculating proportional amounts of the different sugars.

Soxhlet's modification of Fehling's solution is used for all the official methods except Allihn's method for dextrose, in which case Allihn's modification of Fehling's solution is used. The concentration of the copper solution is the same for both the modifications as for the original Fehling solution, viz, 34.64 grams of pure crystallized copper sulphate per 500 cc. Fehling's alkaline tartrate was made by using 55 to 64 grams of sodium hydrate and 173 grams of Rochelle salts per 500 cc, while Soxhlet's modification requires 50 grams of sodium hydrate and Allihn's modification 125 grams of potassium hydrate per 500 cc. In my experience Fehling's solution when made up with sodium hydrate gives a more satisfactory precipitation than when potassium hydrate is used, and this alkali is much more commonly employed than potassium hydrate. Glendenning[a] has studied the effect of the two alkalies, when used in equivalent amounts, upon the quantity of copper reduced, and he finds that both dextrose and invert sugar give practically the same reducing power with the two alkalies. Maltose, on the other hand, shows a reducing power of 62 with soda solution and 65 with potash solution. Kjeldahl[b] has studied the effect of a variation of the strength of the soda solution upon the reduction, and finds that between limits of one-half and double the normal weight of soda solution the variation was not more than 1 per cent with dextrose and reducing sugar, while with lactose and maltose there was a wide variation, as the following table shows:

TABLE I.—*Effect of varying soda solutions upon reduction.*

Kind and weight of sugar.	Soda solution used.	Copper reduced.
		Mg.
150 milligrams lactose	50 cc solution containing 130 grams NaOH per liter	198.4
	50 cc solution containing 65 grams NaOH per liter	207.2
	50 cc solution containing 32.5 grams NaOH per liter	214.7
360 milligrams maltose	50 cc solution containing 50 grams NaOH per liter	411.7
	50 cc solution containing 78 grams NaOH per liter	396.9
220 milligrams dextrose	50 cc solution containing 50 grams NaOH per liter	412.6
	50 cc solution containing 78 grams NaOH per liter	411.0

There seems to be no reason why the copper and alkaline tartrate solutions should not be of the same composition for the various sugars, since the formula must of necessity be an empirical one.

The conditions under which the reduction takes place are of particular importance, and much of the work this year has consisted in a study of these conditions. Recently a number of valuable papers have been published that have a direct bearing upon this subject, and results of some of these investigations will be given as a part of this report.

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[a] Jour. Chem. Soc., 1895, p. 999.

[b] Compte-rendu des travaux du laboratoire de Carlsberg, 1895.

EFFECT OF TIME AND MANNER OF HEATING.

O'Sullivan [a] was the first to carry on the reduction in a boiling-water bath instead of heating directly over a free flame; and this method is now quite generally used among English chemists. Brown, Morris, and Millar [b] have determined the cupric reducing power of dextrose, invert sugar, and levulose by heating at the temperature of boiling water for twelve minutes. These values are considerably higher than when the reduction is made by boiling over a free flame for two minutes.

Kjeldahl [c] in an elaborate series of experiments has determined the copper reducing power of dextrose, invert sugar, levulose, maltose, lactose, and galactose by carrying on the reduction in an atmosphere of hydrogen, and continuing the time of heating for twenty minutes from the time the mixed solutions are placed in the water bath. The following table of compiled data shows the reducing values for dextrose and invert sugar obtained by the three different methods of manipulation:

TABLE II.—*Reducing values for dextrose and invert sugar obtained by three different methods.*

Sugar.	Dextrose.			Invert sugar.		
	Copper by boiling 2 minutes. [a]	Copper by heating in water bath 12 minutes. [b]	Copper by heating in water bath 20 minutes. [c]	Copper by boiling 2 minutes. [d]	Copper by heating in water bath 12 minutes. [b]	Copper by heating in water bath 20 minutes. [c]
Mg.	*Mg.*	*Mg.*	*Mg.*	*Mg.*	*Mg.*	*Mg.*
50	98.2	103.0	111.4	96.0	97.5	105.6
75	147.2	154.3	163.2	142.8	146.8	155.6
100	195.0	202.7	212.5	188.8	194.1	203.4
125	242.0	249.6	259.2	233.2	240.0	249.0
150	288.2	293.4	303.3	276.8	283.2	292.3
175	333.4	335.0	345.0	318.8	325.1	333.7
200	377.5	374.5	384.0	360.4	365.0	372.7

[a] Figures taken from Allihn's table for dextrose.
[b] Figures taken from tables by Brown, Morris and Millar.
[c] Figures taken from Kjeldahl's tables.
[d] Figures taken from Meissl's table for invert sugar.

The values obtained by Kjeldahl are much higher than those obtained by either of the other methods, owing partially to the longer time of heating, but principally to the fact that the reduction was made in an atmosphere of hydrogen, and consequently no oxidation took place during the period of heating.

I have also conducted a series of experiments with dextrose and invert sugar in which the time of heating in a water bath was varied between five minutes and twenty minutes. The following are the results:

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[a] Jour. Chem. Soc. Trans., 2: 125 (1876).
[b] Jour. Chem. Soc. Trans., 1897, p. 275.
[c] Compte-rendu des travaux du laboratoire de Carlsberg, 1895.

TABLE III.—*Copper reduced upon heating in boiling water bath for varying periods.*

Sugar.	Manner and time of heating.	Copper reduced.	Increase in copper for 5-minute periods.
Dextrose (milligrams).		*Milligrams.*	*Milligrams.*
150.3	Boiled 2 minutes	286.5	....
150.3	Heated in boiling water[a] 5 minutes	281.0	....
150.3	Heated in boiling water[a] 10 minutes	286.5	5.5
150.3	Heated in boiling water[a] 15 minutes	289.4	2.9
150.3	Heated in boiling water[a] 20 minutes	292.8	3.4
Invert sugar (milligrams).			
162.8	Boiled 2 minutes	303.5	....
162.8	Heated in boiling water[a] 5 minutes	298.4	....
162.8	Heated in boiling water[a] 10 minutes	303.2	4.8
162.8	Heated in boiling water[a] 15 minutes	307.1	3.9
162.8	Heated in boiling water[a] 20 minutes	310.5	3.4

[a] The sugar was added to the cold Fehling solution, and time was counted from the moment the mixture was placed in the boiling water bath.

The above figures show that the method of boiling for two minutes over a direct flame gives results practically identical with heating for ten minutes in a boiling-water bath.

One advantage claimed for the method of heating in a boiling-water bath is that after heating for 10 to 15 minutes the reduction is practically completed, and upon further heating the amount of reduction that takes place is exceedingly small. Table III shows that this is not the case, but that after fifteen minutes' heating five minutes additional heating gave 3.4 milligrams of copper. Kjeldahl found in his work that upon heating for twenty minutes there was a material spontaneous precipitation of copper, which greatly augmented when the time of heating was prolonged. The following table shows results obtained by myself when the period of boiling over a direct flame was varied between just bringing to a boil and four minutes' boiling:

TABLE IV.—*Copper reduced upon boiling over free flame for varying periods.*

Sugar.	Time of heating.	Copper precipitated.	Increase in copper.
Dextrose (milligrams).		*Milligrams.*	*Milligrams.*
250	Brought to boil	437.2	....
250	Boiled 2 minutes	439.5	2.3
125	Brought to boil	227.9	....
125	Boiled 2 minutes	229.8	1.9
Invert sugar (milligrams).			
185.9	Brought to boil	245.1	....
185.9	Boiled 1 minute	346.8	1.7
185.9	Boiled 2 minutes	347.6	0.8
185.9	Boiled 3 minutes	348.7	1.1
185.9	Boiled 4 minutes	350.4	1.7

The increase in the amount of copper reduced for each additional minute's boiling is higher somewhat than when heating in boiling water is employed, but the difference is not sufficient to give any preference to either method.

In an effort to verify Kjeldahl's statement that appreciable amounts of copper are precipitated upon heating the Fehling solution, I found that only traces were precipitated when the solution was heated for twenty minutes in boiling water. Heating over a direct flame gave no reduction upon two minutes' heating, 2 milligrams upon four minutes, 5.6 milligrams upon six minutes, and 8.5 milligrams upon eight minutes' boiling. However, different solutions undoubtedly vary in this respect, depending upon the length of time the alkaline tartrate solution has been made up. In all cases, whatever method is used, the amount of copper precipitated spontaneously should be determined and deducted from the amount obtained in the sugar determinations.

EFFECT OF SURFACE EXPOSED DURING REDUCTION.

But few if any of the reducing sugar methods prescribe a receptacle of definite size for the reduction. While beakers of varying sizes are generally employed, Erlenmeyer flasks, and even porcelain dishes, are occasionally used. Kjeldahl has studied the effect upon the amount of copper reduced of the surface of liquid exposed when dishes of different sizes are used for the reduction, and he gives the following interesting results:

TABLE V.—*Effect of amount of surface exposed to the air upon the copper reduced.*

Sugar.	Surface exposed to air	Copper reduced.	Difference.
Milligrams.	*Sq. cm.*	*Milligrams.*	*Milligrams.*
60	(a)	126.3	....
60	2	126	0.3
60	17	123.9	2.4
60	21	121.5	4.8
60	65	114.8	11.5
60	186	106.6	19.7

[a] None; reduction in hydrogen.

In all the experiments of Kjeldahl the time of heating was twenty minutes. I have conducted a similar series of experiments, the results of which are given below in Table VI:

TABLE VI.—*Effect of surface exposed to the air upon the copper reduced under varying conditions of manipulation.*

Invert sugar.	Receptacle used.	Surface exposed.	Manner and time of heating.	Copper reduced.	Difference.
Mg.		*Sq. cm.*		*Mg.*	*Mg.*
150.5...	Erlenmeyer	(a)	Boiled 2 minutes	284.2	....
150.5...	.....do	(b)	.....do	284.5	....
150.5...	Beaker	40	.....do	281	3.4
150.5...	Erlenmeyer	(a)	Heated 15 minutes in boiling water.	291.7	....
150.5...	.....do	(a)	.....do	290.6	....
150.5...	Beaker	40	.....do	286.7	4.5
168.5...	Erlenmeyer	26.5	Boiled 2 minutes	313.7	....
168.5...	Small beaker	26.5	.....do	313.9	....
168.5...	Beaker	40	.....do	313.4	.5
168.5...	Erlenmeyer	26.5	Heated 15 minutes in boiling water.	320	....
168.5...	Small beaker	26.5	.....do	320.1	....
168.5...	Beaker	40	.....do	317.2	2.8

[a] None; reduced in CO_2.

[b] None; reduced in hydrogen.

It is seen from Tables V and VI that the greater the surface of liquid exposed to the air during the reduction the less is the amount of copper reduced. In other words, the oxidation of the cuprous oxid by exposure to the air is very marked. In Kjeldahl's series of experiments the amount of copper varied by an extreme difference of 19.7 mgs with receptacles ranging in surface exposed from none to 186 sq. cm. While it is hardly probable in ordinary work that the reduction would be made in a dish giving an exposed area of 186 sq. cm., it is undoubtedly true that dishes giving from 15 to 65 sq. cm exposed surface are frequently employed. I have found that a beaker 7.5 cm in diameter is of very convenient size for making the reduction, and have used this size in all my regular work. It appears from Table VI that the amount of oxidation is greater when the reduction is conducted in a boiling-water bath, heating for fifteen minutes, than when the reduction is made by boiling over a free flame for two minutes. In the first series the amount of oxidation is greater by 1.1 mg. when reduced in boiling water for fifteen minutes; and in the second series the amount of oxidation is greater by 2.3 mg of copper when heated in boiling water for fifteen minutes, when the surface exposed is increased from 26.5 to 40 sq. cm. The method employed by Kjeldahl of conducting the reduction in an atmosphere of hydrogen undoubtedly gives more nearly the true reduction than any other method yet employed, but the inconveniences attending such a manipulation would preclude its general use.

DETERMINATION OF COPPER BY WEIGHING THE CUPROUS OXID.

The determination of copper by direct weighing of the cuprous oxid was first suggested by O'Sullivan and Stern,[a] who state that results obtained by this method are as accurate as given by the methods ordinarily used. In 1900 Knight reported to this association results of a series of experiments showing that this method of determining the copper checks up very closely with the electrolytic method. It has been our experience in the laboratory of the Bureau of Chemistry, where we have practiced direct weighing of the cuprous oxid for the past two years, that the method gives reliable results, and that the determination is much more easily made than by any of the official methods of this association. I have made a number of determinations checking this method with the electrolytic method, results of which are given in Table VII.

TABLE VII.—*Comparison of the method of direct weighing of cuprous oxid with electrolytic method.*

Weight of copper.		Difference in weight.	Weight of copper.		Difference in weight.
By weighing as Cu_2O.	By electrolysis.		By weighing as Cu_2O.	By electrolysis.	
Mg.	*Mg.*	*Mg.*	*Mg.*	*Mg.*	*Mg.*
440.0	439.6	-0.4	319.9	320.1	+0.2
437.2	437.8	+ .6	316.7	317.2	+ .5
227.9	229.1	+1.2	317.7	316.5	-1.2
229.5	230.9	+1.4	284.9	284.0	- .9
447.7	448.0	+ .3	282.2	281.0	-1.2
447.3	447.8	0.0	290.0	288.8	-1.2
320.5	321.4	+ .9	286.9	286.9	0.0
278.4	278.3	- .1	287.8	288.0	+ .2
279.2	280.6	+1.4	303.6	303.9	+ .3
278.0	278.2	+ .2	303.4	303.3	- .1
233.6	235.4	+1.8	289.5	289.5	0.0
460.4	460.5	+ .1	298.3	298.8	+ .5
459.5	460.3	+ .8	303.2	303.7	+ .5
316.7	317.2	+ .5	303.2	302.4	- .8
312.1	313.4	+1.3	Average..	....	+ .22

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[a] Jour. Chem. Soc. Trans., 1896, p. 1691.

Upon 29 determinations the electrolytic method gives results averaging 0.22 mgs higher than by weighing as cuprous oxid, and the differences by the two methods vary between −1.2 and +1.8 mgs. A small amount of asbestos is dissolved by the hot Fehling solution during filtration, and in the recorded experiments this factor was not taken into consideration. If the asbestos is carefully prepared by first digesting with 1:3 hydrochloric acid and then with Fehling's solution, the amount of asbestos dissolved upon filtering the hot Fehling solution need not exceed 0.3 to 0.5 mgs. Should this correction be added to results obtained by weighing the cuprous oxid, the two methods would check very closely. I have found it convenient to dissolve off the cuprous oxid with hot nitric acid after each determination, and in this way the same felt of asbestos may be used several times.

The method of weighing the cuprous oxid is conducted as follows: Load a Gooch crucible with a film of asbestos about one-fourth of an inch thick; wash thoroughly with water to remove small particles of asbestos, then with about 10 cc of alcohol, and finally with 10 cc of ether. Dry in a water oven at 100° C. for twenty-five to thirty minutes, and cool for one-half hour before weighing. After filtering the cuprous oxid into the Gooch and washing thoroughly with water, the precipitate and Gooch are washed with 10 cc of alcohol and then with 10 cc of ether as in the preparation of the Gooch. Dry for twenty-five to thirty minutes in water oven at 100° C.; cool for one-half hour and weigh. The weight of cuprous oxid multiplied by 0.888 gives weight of metallic copper. It has been my experience that twenty-five to thirty minutes heating at 100° is sufficient to thoroughly dry the precipitated cuprous oxid and asbestos without danger of the suboxid undergoing oxidation, providing the washing with alcohol and ether has been thorough.

Mr. **Wiley.** I would like to suggest that Mr. Gudeman's paper on the "Determination of glucose," which was to be submitted to the association, be returned to him for further elaboration, and that after such modification it be incorporated in the proceedings of this meeting.

The **President.** If there is no objection, the paper will be returned to Mr. Gudeman for further consideration and then be included in the proceedings. The secretary is so instructed.

THE DETERMINATION OF GLUCOSE.

By Edward Gudeman, *Chicago, Ill.*

The method used for the determination of commercial glucose is the one given in the provisional methods of the Association Official Agricultural Chemists, 1901. The method consists in polarizing before and after inversion, calculating the per cent of sugar "S" by Clerget's formula and inserting this value in the following formula:

$$G=\frac{(a-S)\,100}{175}.$$

G=Commercial glucose.
a=Direct polarization.
175=Polarization factor for commercial glucose.

The use of this factor (175), or any other factor between 80 and 198, will give incorrect results, a correct result being purely accidental. It is only in laboratory work that one has the same substance to experiment with that has been used in making up the mixtures under examination and on which a correct factor for that special glucose or grape sugar can be determined.

One is often at sea as to what is meant by the term commercial glucose. In England the terms glucose and grape sugar are synonymous, the sirup being designated as liquid glucose. In America the term glucose refers only to the sirup, the solid being designated as grape sugar. The reducing substances of glucose and grape sugar are commercially, but incorrectly, known as "dextrose" and the nonreducing substance as "dextrins." The composition of commercial glucose and grape sugar varies between wide limits, the extremes being about as follows:

Glucose {Reducing substance 40 to nonreducing substance 60; ratio 6 : 9.
Reducing substance 55 to nonreducing substance 45; ratio 11 : 9.

Grape sugar {Reducing substance 80 to nonreducing substance 20; ratio 36 : 9.
Reducing substance 92 to nonreducing substance 8; ratio 103.5 : 9.

Glucose is a thick sirup, 41° to 45° Baumé, containing 13 to 22.5 per cent of water. Grape sugar is a solid, 41 to 45° Baumé, containing 11 to 21.0 per cent of water. The old standard Baumé scale is still used in this industry, and the determinations are made at 140° F., results reported at 100° F., a correction of 1 degree being made for every 40° F. The standard accepted by the manufacturers and the trade is sp. gr. 1.41152=42° Baumé.

The conversion of starch with acids gives the commercial products known as dextrin, glucose, grape sugar, and anhydrous grape sugar (dextrose). Starch and its conversion products do not contain over 1 per cent impurities, and, as these have little or no effect on the polarization nor on the copper reduction, they have not been taken into consideration. All results are calculated on the sum of the reducing and nonreducing substances being equal to 100. All results are figured on the amount of dry substance contained in the mixtures.

If the conversion of starch with acid is carried to a point where a dilute iodin solution will just give a distinct color reaction, we have glucose; continued to where 95 per cent alcohol gives a faint cloud, hardly a precipitate, we have grape sugar containing about 85 per cent reducing substance. For still higher converted sugars a time factor must be introduced. Carried beyond a given point a back conversion takes place, with strong decomposition and loss of purity.

The ratio of the reducing substance to nonreducing substance depends on the accuracy in stopping the conversion, for neutralization, at the exact point decided upon. This ratio determines whether the product is glucose or grape sugar, and no sharp dividing line exists. The rotating powers of glucose and grape sugar depend absolutely on this ratio. Actually no two batches of commercial glucose or grape sugar are identical; for all practical purposes they are alike, as a few points either way from the standard decided upon will make no difference in the appearance, taste, or working qualities of these products.

The conversion of starch does not take place in direct steps; immediately when the starch is liquefied it passes into the various conversion products. At no point of the conversion is a single product, either of dextrin or dextrose obtained. The rotation of dextrin and dextrose can easily be determined on the pure products separated from the mixture. At no point of the conversion do you obtain the extreme polarization figures, 230 for maize dextrin, and 80 to 153 for maize dextrose. The accompanying graphic chart (fig. 1) shows the polarization and the percentage of nonreducing and reducing substances for the following commercial products: Glucose, grape sugar, dextrose, and chemically pure dextrose.

The first commercial product in the acid conversion is glucose: Line ab: Reducing substance 40 to nonreducing substance 60. Line abc: Reducing substance 55 to nonreducing substance 45.

The second product is grape sugar: Line abcd: Reducing substance 80 to nonreducing substance 20. Line abcde: Reducing substance 92 to nonreducing substance 8.

The final commercial product, anhydrous grape sugar (dextrose), passes through the glucose and grape-sugar stages: Line abcdefg to abcdefgh. Dextrose is birotary

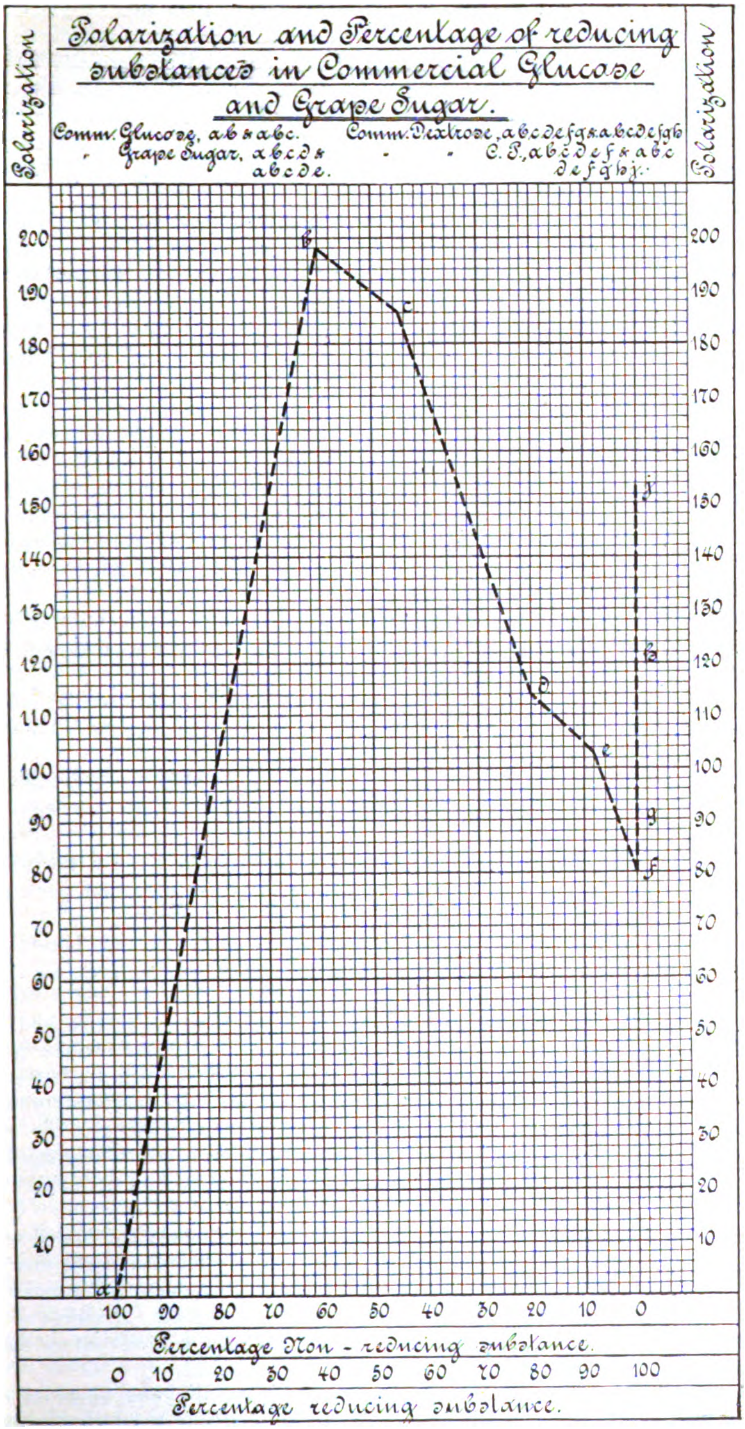

FIG. 1.—Polarization of commercial glucose and grape sugar and percentage of reducing substances therein.

and as the rotation of the commercial product is higher than that of grape sugar, there is no doubt that it must have passed through the lowest point for dextrose before going up again. The high point is only obtained on specially prepared chemically pure dextrose. The rotation of dextrose is birotary, the low point being reached after a comparatively long time in cold solution, but more rapidly on heating to boiling. Under these conditions it is not possible to have a high rotating dextrose in the starch conversion liquor, as the same has been exposed to high temperature, being converted under steam pressure.

The provisional methods state that commercial glucose used for mixing with jams and jellies polarizes at 150, that used for chewing gums at 185, and that used with honeys, sirups, and molasses at 175, and it is suggested to use these values when examining such products. I have never known the manufacturers of glucose to make special grades for these purposes, except for the confectioner, and this grade differs only from the regular glucose by the larger amount of acid salt it contains (bisulphite of soda). The use therefore of different factors of rotation, subject to the samples under examination, will lead to greater errors than the use of a constant (175), even if the same is not correct. Depending on what factor is used, a mixture containing 20 per cent commercial glucose can show from 14.4 to 27.7 per cent, a possible error of 35 per cent. Commercial grape sugar rotating 85 to 190 will show from 43.5 to 225 per cent, depending on the factor used, a possible error of 125 per cent. The addition of cane sugar or invert sugar to such mixtures can cause the polarization figures to become so abnormal, as to be hardly a qualitative indication of the presence of glucose or grape sugar.

On mixtures of which the percentage composition was known, as also the correct rotating power of the component parts, the following results were obtained, applying the formula given in the provisional methods, 1901:

I. With a mixture containing grape sugar 10 per cent, invert sugar 25 per cent, and cane sugar 65 per cent, $a=65$ and $b=-22$. Applying the formula, we find that the grape sugar=0 per cent, an error of 100 per cent.

II. With a mixture containing grape sugar 28.5, invert sugar 6.5, and cane sugar 65 per cent, $a=87$ and $b=0$. By the formula grape sugar=25.8 per cent, an error of 9.6 per cent.

III. With a mixture containing grape sugar 15, invert sugar 20, and cane sugar 65 per cent, $a=71$ and $b=-16$. Applying the formula, grape sugar=7.1 per cent, an error of 52.7 per cent.

IV. With a mixture of 40 per cent grape sugar and 60 per cent invert sugar, $a=13.6$, and $b=13.6$. Applying the formula, grape sugar=17 per cent, an error of 57.5 per cent.

Similar results were obtained with glucose and mixtures of glucose and grape sugars. These errors were found when using the correct factor for the rotating power of the commercial grape sugar. When the correct polarization factor is not known, the errors are increased or decreased, and in some cases may exactly balance each other, when accidentally the application of the formula will give the correct result. This is not a theoretical consideration, as it has a direct bearing on the analyses of sirups, honeys, preserves, jams, candies, molasses, wines, maple sugars, and similar saccharine food products.

Plus polarization before and minus after inversion is generally considered as indicating the absence of commercial glucose or grape sugar. This is not necessarily the case, Mixtures I and III showing plus 65 and 71 before inversion, and minus 22 and 16 after inversion, with 10 to 15 per cent grape sugar present. Mixtures of commercial glucose or grape sugar with invert sugar can show a direct minus reading, increased strongly after inversion if cane sugar is also present.

The quantitative determination of commercial glucose or grape sugar can easily and quickly be made without use of the polariscope and variable factor. If invert sugar is present, the same must be determined by double polarization. All results are calculated on the basis of the dry substance found in the mixture.

In mixtures free from invert sugar:

I. Determine total directly reducing sugars=a (from glucose or grape sugar).

II. Determine cane sugar by reduction after inversion at 67° C.=b (or Clerget's method calculating reducing power=b).

III. Invert with malt and determine total reducing sugars=c (as in determination of starch).

$c-(a+b) \times 0.932=d$.

d=nonreducing substance from glucose or grape sugar.

a+d=glucose or grape sugar, depending on ratio of a:d.

If invert sugar is present, it must be determined by double polarization at 20° and 87° C.=i.

a−i=A, reducing substance from glucose or grape sugar.

$c-(a+b+i) \times 0.932=d$.

A+d=glucose or grape sugar, depending on ratio of A:d.

The error due to use of factor 0.932 is very small—not over 1 per cent—on a pure low converted glucose containing 60 per cent nonreducing substance. If the mixtures contain starch, same must be separated by cold filtration. Starch in solution will be determined and reported as part of the nonreducing substance. The iodin test will show the presence of starch.

Results by this method are generally low, due, no doubt, to the decomposition of cane or invert sugars present. The larger the percentage of glucose or grape sugar in the mixtures the more accurate the results. The error is never over 3 per cent of the amount of glucose or grape sugar present, averaging about 1 per cent. Duplicates will agree to within 0.2 per cent.

If the mixtures contain no cane nor invert sugars, the malt treatment can be replaced by a direct acid conversion at high temperature. This is not advisable, as other substances (fiber, gums, etc.) will be partly converted into reducing substances. Mixtures of glucose with grape sugar will be indicated by the abnormal ratio of a or A:d being between 11 and 36:9, but for all practical purposes such mixtures can be considered as high converted glucose or low converted grape sugar.

CLERGET'S METHOD AND MODIFICATIONS.

By L. M. Tolman.

At the twelfth annual meeting of this association, Mr. Ewell, then reporter on sugar, recommended that the Clerget and Lindet methods for determination of sucrose be dropped as official methods, and that the German official method, at that time one of the official methods, be made the official method of this association. For some reason not given in the proceedings this was not done, but the German method and the Lindet methods were dropped as official methods and the Clerget retained. The reporter on sugar at the seventeenth annual meeting recommended that a study be made of the Clerget and German methods. That has, therefore, been the line of work attempted by the associate referee this year.

The Clerget method, as now used by this association, is a modification of the original method proposed by Clerget, in that 26.048 grams of sucrose dissolved in 100 cc polarize 100 degrees on the sugar scale, while Clerget used the French normal weight, then 16.471 grams per 100 cc. In every other particular the original method is followed, and the factor determined by Clerget is used.

When Clerget's method is exactly followed and 16.471 grams of sugar are used, his results are correct, and his factor 144 should be used; but when 26.048 grams of sugar are used this factor is too low, owing to the increased concentration. Ross reported at the ninth annual meeting of this association as high a factor as 145.2, using 26.048

grams of sugar, but inverting in a slightly different manner from that prescribed by Clerget. Bornträger[a] obtained as high a factor as 134.98 at 20° C., using 26.048 grams, and, following Clerget's method exactly with this exception, he obtained the factor 144.65, showing that the factor of Clerget is too low for the official method.

Of late years very little has been done to determine exactly the proper factor, all of the investigations having been conducted along the lines of the German method. Herzfeld[b] found that the factor regularly increased in ratio with the increased concentration. The factor determined for 1 gram sugar in 100 cc was 141.85, if the German official method was used in inversion, and it increased to 142.66 for 13 grams of sugar, 143.13 for 20 grams, and to 143.94 for 26 grams. From this investigation Herzfeld derived the following formula for determining the factor for any concentration:

$$S = \frac{(A—B)\ 100}{141.85 + 0.05\ B - \frac{T}{2}}$$

S = sucrose
A = direct polarization
B = invert polarization
T = temperature.

To 141.85, which is the factor for 1 gram of sugar in 100 cc, there is added 0.05 of the invert reading. This formula applies only when the Herzfeld method of inversion is followed. When the official method of this association is followed with the increased amount of acid (10 cc to 110 cc), the increase in the factor due to concentration is larger.

In a paper published in the Journal of the American Chemical Society for June, 1902, I showed that the increment of the factor was 0.062 of B, or the invert reading. This would make the factor 144.44 for 26.048 grams, giving a variation of from 141.85 to 144.44 for different weights of sugar from 1 gram to 26 grams, while with the German method there is a variation of from 141.85 to 142.66. The latter method, therefore, is more accurate, unless the factor is calculated for each concentration, as proposed by Herzfeld, substituting, however, in his formula 0.062 for 0.05 for the increment of the factor. The reason for this difference in the factor of the two methods is due entirely to the amount of acid used in inversion.

It has been shown by a number of chemists that hydrochloric acid increases the specific rotatory power of levulose. Weber, at the ninth annual meeting of this association, reported that hydrochloric acid increased the reading of a solution of invert sugar, and that this increase varied with the amount of acid. Lippmann[c] gives the following table, showing the effect of different amounts of acid:

TABLE I.—*Effect of different amounts of hydrochloric acid on polarization of invert sugars.*

Weight of sugar.	Polarization, °Ventzke.			
Grams.	*5 cc HCl.*	*10 cc HCl.*	*15 cc HCl.*	*20 cc HCl.*
26.048.....	–34.00	–35.04	–35.95	–36.80
13.024.....	–33.00	–34.12	–35.15	–36.03

Lippmann's polarizations were all made at 20° C., and in the case of the half-normal weight the reading was multiplied by 2 for comparison. This table shows that hydrochloric acid, by its simple presence, affects the polarization.

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[a] Ztsch. Rub. Zuck. Ind., 40: 898 (1890).

[b] Ztch. Rub. Zuck. Ind., 40: 194 (1890).

[c] Chemie die Zucherartin, p. 800.

In Table II are some results of my own, showing that the addition of acid in the cold had the same effect as when it was used in the process of inversion:

Table II.—*Effect of different amounts of hydrochloric acid on polarization of invert sugars when added in the cold.*

Description of sample.	Polarization.	Temperature.	Change in rotation.
	° *Ventzke.*	°C.	
No. 1, containing no HCl	−23.0	18.0	....
No. 2, containing 5 cc HCl, 67° C	−24.2	18.4	1.2
No. 3, containing 10 cc HCl, 67° C	−25.0	17.8	2.0
No. 4, containing 5 cc HCl (cold)	−24.2	18.4	1.2
No. 5, containing 10 cc HCl (cold)	−25.0	18.0	2.0
No. 6, containing 10 cc HCl (N/4) (cold)	−23.4	18.0	0.4

Table I also shows the different effects of equal amounts of acid on different weights of sugar. This is further shown in fig. 2, which illustrates the action of 10 cc of acid in 110 cc of sugar solutions of varying degrees of concentration.

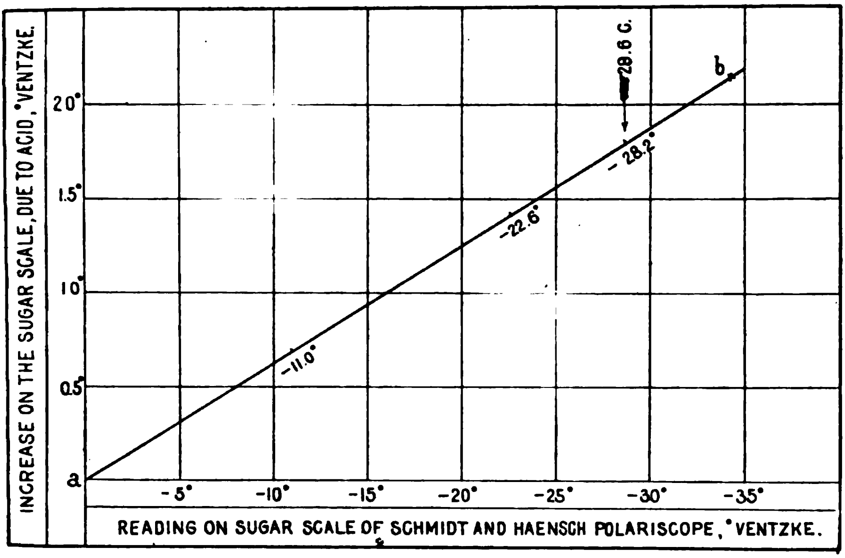

Fig. 2.—Effect of 10 cc of hydrochloric acid on 110 cc of sugar solutions of varying concentration.

To obtain this diagram (fig. 2) solutions of invert sugar of varying strengths were prepared and 10 cc of acid were added to 110 cc of the solution. The difference between the reading of the solution before and after the addition of the 10 cc of acid would give a point on the line a–b. The diagram also shows the effect due to concentration of the solution plus the effect due to the acid.

This work demonstrates that the factor for the Clerget or any other formula is a variable and that the amount of the variation depends on both the amount of acid and the amount of sugar used. The smaller the amount of acid, the more nearly the factor approaches a constant. These two facts, combined with the consideration that the factor for the German method has been carefully determined and that all the investigations of late years have been conducted according to this method, emphasize the wisdom of its adoption as the most accurate method now available.

At the suggestion of Dr. Spencer, the associate referee made some investigations as to the inversion of sugars at ordinary temperatures. This would have several advantages over the methods now employed. First, there would be no danger of the destruction of levulose as occurs from overheating. Further, when a large volume of work is being carried on it would be convenient to set the solutions aside and allow them to invert while other work is being done.

Hammerschmidt[a] worked out the time necessary for the complete inversion of 13.024 grams of sugar at different temperatures and with varying amounts of hydrochloric acid. The following table shows his results for temperatures ordinarily prevailing in the laboratory:

Table III.—*Time necessary to invert 13.028 grams sucrose dissolved in 100 cc (Hammerschmidt).*

Temperature.	5 cc HC.[a]	10 cc HC.[a]
°*C.*	*Hours,*	*Hours.*
10	225.0	94.0
15	101.0	44.0
20	47.0	20.0
25	23.0	10.0
30	11.6	5.0

[a] Specific gravity, 1.18.

From these figures it will be seen that with a temperature of 25° C. and 10 cc of hydrochloric acid to 100 cc, complete inversion takes place in 10 hours. Borntrãger[b] showed that complete inversion could be brought about at from 20° to 23° C. by standing from eighteen to twenty hours.

The associate referee has made a number of tests of this method, conducting the work along the lines of the German official method. To a solution of 13.024 grams sucrose, 50 cc water and 5 cc hydrochloric acid (sp. gr. 1.18) were added. The solution was made up to 100 cc, placed in a 200-mm tube and the reading made. The results were as follows:

Table IV.—*Process of inversion during twenty-four hours (German official method).*

Time.	Polarization.	Temperature.	Time.	Polarization.	Temperature.
	° *Ventzke.*	°*C.*		° *Ventzke.*	°*C.*
11 a. m	+50.0	25.8	3.10 p. m	− 7.20	25.8
12.40 p. m	+10.0	25.8	3.30 p. m	− 8.20	26.0
12.44 p. m	+ 9.50	25.8	3.57 p. m	− 9.50	26.0
12.55 p. m	+ 7.60	25.8	4.24 p. m	−10.60	26.5
1.15 p. m	+ 4.60	25.8	5 p. m	−11.65	27.3
1.34 p. m	+ 2.00	25.8	8 p. m	−14.30	27.0
1.48 p. m	.30	25.8	11.45 p. m	−14.85	26.4
2 p. m	− 1.50	25.8	9.05 a. m	−15.30	25.0
2.32 p. m	− 4.30	25.8			

Ztsch. Rub. Zuck. Ind. **40:** 465 (1890).

[b] Ztschr. Rub. Zuck. Ind. **40:** 881 (1890).

Borntrāger[a] obtained very similar results at a temperature from 20 to 22° C. In order to determine whether the factor used in the German official method would be correct for this method of inversion, a number of comparative determinations were made on the same sugar, using both methods. The results are shown in the following table:

TABLE V.—*Comparison of inversion in the cold and by the German official method (half normal weight).* [a]

INVERSION IN THE COLD.

Polarization.	Temperature.	Cor. to 20° C. (× 2).	Sodium acetate added.
° *Ventzke.*	°*C.*	° *Ventzke.*	*Grams.*
−15.28	24.5	−32.81	....
−15.32	24.6	−32.94	....
−15.27	24.7	−32.89	....
−14.65	27.1	−32.85	1
−14.60	27.1	−32.75	3
−14.56	27.2	−32.75	....
−14.68	26.8	−32.76	2
−15.50	23.6	−32.80	2
−15.38	23.6	−32.56	4

GERMAN OFFICIAL METHOD.

Polarization.	Temperature.	Cor. to 20° C. (× 2).	Sodium acetate added.
−15.90	22.05	[b] −32.83	....
−15.78	22.65	[b] −32.89	....
−15.70	22.95	[b] −32.88	....

[a] Five cc hydrochloric acid (sp. gr. 1.18) were added to each solution which stood twenty hours at a temperature of 20° C.

[b] Average, −32.86 = 142.86 factor.

These solutions were polarized in a 400-mm tube to decrease the error of reading, and at exactly the same temperature the average of the factors calculated from inversion in the cold was 142.88, and by inversion by the German official method, 142.86, which shows a very satisfactory agreement between the two methods.

From the table it will also be seen that the presence of considerable amounts of sodium acetate does not interfere with the inversion, although it probably reduces the invert polarization, as is shown from the factor 142.85 when 1 gram of sodium acetate is present and 142.56 when 4 grams are present. Herles[b] showed that an addition of 3.4 grams sodium acetate reduced the reading of an invert sugar solution, inverted with hydrochloric acid, from −33.02° to −32.86.°

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[a] Ztschr. Rub. Zuck. Ind. **40:** 881 (1890).

[b] Ztschr. Rub. Zuck. Ind. **40:** 220 (1890).

When the normal weight of sugar (26.048 grams) was used, the comparative inversions showed practically the same agreement, as can be seen from the following table:

TABLE VI.—*Comparative inversions made in the cold and by the German official method (normal weight).*[a]

INVERSION IN THE COLD.

Polarization.	Temperature.	Cor. 20° C.	Citric acid added.
° *Ventzke.*	° *C.*	° *Ventzke.*	*Grams.*
—30.73	26.2	—33.83	....
—30.78	26.2	—33.88	....
—30.83	26.1	—33.96	....
—30.78	26.1	—33.83	....
—30.78	25.9	—33.63	....
—30.75	25.9	—33.60	2
—32.30	23.0	—33.80	2

GERMAN OFFICIAL METHOD.

—32.85	22.4	[b]34.00	....
—32.32	23.2	[b]33.92	....
—32.27	23.4	[b]33.97	....

[a] Five cc hydrochloric acid (sp. gr. 1.18) were added to each solution which stood from twenty to wenty-four hours at a temperature of 26° C.

[b] Average, —33.96.

The factor as calculated from inversion in the cold is 143.86 and from inversion by heating 143.96. The method employed was to dissolve 13.024 or 26.048 grams in a 50-cc solution, add 5 cc hydrochloric acid, allow to stand at room temperature from twenty to twenty-three hours, make up to 100 cc, and polarize. This method requires that the temperature shall be above 20° C., as can be seen from Table III (Hammerschmidt). At lower temperatures the time of inversion is too long. At a temperature of 20° to 24° C., following the method as given above, the complete inversion of 13.024 grams of sugar will take place in from ten to twenty hours; at 25° it only takes ten hours. In many localities this is the temperature of the laboratory for most of the year, and it can be easily obtained in any laboratory.

Following Clerget's method as to the amount of acid and inverting in the cold with the normal weight of sugar (26.000 grams to 100 true cc) a polarization of —33.99° at 20° C. was obtained. When 10 cc of hydrochloric acid were added to 100 cc with the normal weight of sugar, and inversion was effected by standing twenty to twenty-four hours at 26° C., the polarization was —34.23° at 20° C. The addition of 2 grams of citric acid did not affect the reading.

In many kinds of work it would be a decided advantage to have an inverting agent which did not affect the specific rotatory power of the levulose. This is seen in the polarization of honey and the estimation of sucrose by inversion as set forth in the following table on the polarization of honeys:[a]

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[a] U. S. Dept. of Agr., Division of Chemistry Bul. 13, Pt. 6.

TABLE VII.—*Polarization of honeys.*

		° Ventzke.	
Direct.	Invert.	Cane sugar.	Calculated invert reading.
−20.00	−20.90	0.72	−21.25
−17.60	−18.32	.93	−18.59
−16.8	−18.40	1.23	−18.20
−15.8	−17.5	1.31	−16.78
−11.0	−12.0	....	−11.68
−13.2	−15.6	....	−14.00
−16.50	−18.00	....	−17.52
−14.80	−15.60	....	−15.70
− 9.40	−10.67	.96	− 9.98
−12.90	−13.86	.73	−13.70
−14.40	−15.40	.76	−15.39
−11.30	−12.65	.99	−12.00
−11.50	−12.65	.85	−12.21
−13.50	−14.00	.30	−14.33
− 9.5	−10.00	.30	−10.05
−11.0	−12.00	.70	−11.68
−10.0	−11.00	.70	−10.62
−11.0	−12.00	.70	−11.68

By the ordinary method of inversion all of the samples appeared to contain cane sugar; but when the change in polarization, due to the addition of the acid, is calculated, as is shown in the fourth column, it can be seen that a considerable number contained no cane sugar whatever.

If an inverting agent could have been used which did not increase the specific rotatory power of levulose there would have been no change in polarization after inversion. Of course this difference can be calculated by the formula

$$S=\frac{(A-(B-.062B))}{141.85-\frac{T}{2}}.$$

If A equals (B—.062B), then the change in polarization is due to the acid added in inverting; but this formula only holds good for invert sugar. The presence of glucose renders such a calculation impossible, but it does not prevent a change in polarization, as is shown by the following data:

TABLE VIII.—*Effect of acid on invert sugar in the presence of glucose.*

Solution.	Polarized.	Temperature.
	° Ventzke.	°C.
13.028 grams invert sugar + 25 cc glucose solution	+9.8	21.0
The same + 10 cc hydrochloric acid	+8.9	21.0
The same + 5 cc hydrochloric acid	+9.4	21.0

Thus it is seen that when glucose which contains invert sugar is polarized before and after inversion, there will be a change of polarization even if there is no sucrose present. This condition would be almost always found in honey adulterated with glucose. This error could be prevented by the use of an inverting agent which did not affect the rotatory power of the levulose.

Weber[a] showed that in invert sugar prepared by inversion with acetic acid, the specific rotatory power of the levulose was the same as that of pure levulose, demonstrating that it had no effect on the specific rotatory power; but as acetic acid is a very weak inverting agent, citric acid being about three times as strong, the latter was tried. The strength used in all cases was 2 grams to 100 cc, and the time of heating thirty minutes at the temperature of boiling water. No destruction of invert sugar takes place under these conditions. The following table shows the results obtained by this method:

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[a] Jour. Am. Chem. Soc., 17: 321.

TABLE IX.—*Inversion by citric acid.*

Weight of sugar.	Polarization.	Temperature.	Polarization (Cor. to 0° C.).	Remarks.
	° Ventzke.	*°C.*	*° Ventzke.*	
Normal	−28.95	26.1	−42.00	Average — 41.95.
Do	−28.89	26.0	−41.89	
Do	−28.97	26.0	−41.97	
Do	−29.00	26.0	−42.05	
One-half normal	−28.25	26.3	−41.39	Polarized in 400 mm tube — 41.49 average.
Do	−28.27	26.4	−41.47	
Do	−28.27	26.5	−41.53	
Do	−28.17	26.6	−41.47	
Do	−29.40	26.7	−42.75	5 cc of hydrochloric acid (sp. gr. 1.186) were added before these were made up to volume; average — 42.78.
Do	−29.29	26.9	−42.76	
Do	−29.35	26.9	−42.80	
Do	−29.35	26.8	−42.75	
Do	+40.80	23.3	....	2 grams of sodium acetate were added with the citric acid, and practically prevented inversion.

From these results it can be seen that there is a small difference between the factor for normal weight and one-half normal weight, i. e., 141.95 and 141.49. This change in factor can be corrected by adding 5 cc hydrochloric acid before polarization, and the factor for the German method, 142.66, can be used, as will be seen from the results obtained when this method was followed. It will be noted, however, that sodium acetate greatly interferes with the hydrolysis and in fact almost stops it. This makes the use of citric acid practically of no value in solutions that have to be cleared with lead acetate, which of course greatly limits the value of the method, but in such substances as contain large amounts of invert sugar and small amounts of cane and do not have to be cleared with lead acetate this method will give very accurate results.

In a great many cases it is necessary to estimate cane sugar in the presence of maltose and dextrin. The following table shows the effect of the different methods of inversion on a glucose sirup containing a very high per cent of maltose and dextrin. In all cases the inversion by standing over night, twenty hours, shows less effect than the German official method, or the inversion with citric acid, boiling half an hour.

TABLE X.—*Effect of inversion on glucoses rich in maltose and dextrin.*

POLARIZATION OF SOLUTION MODIFIED AS INDICATED.

Description of sample.	Original solution.	(1) +5 cc HCl at 68° C.	(2) +5 cc HCl 20 hours in cold.	(3) +2 grams citric acid; one-half hour, 100° C.	Decrease in polarization.		
					(1)	(2)	(3)
	° Ventzke.	*° Ventzke.*	*° Ventzke.*	*° Ventzke.*	*° Ventzke.*	*° Ventzke.*	*° Ventzke.*
	+56.20	+55.87	+55.95	....	−0.33	−0.25	....
	+36.66	....	....	+36.12	....	....	−0.54
	+36.69	+36.40	+36.64	+36.28	.29	− .05	− .41
XXX	+47.15	....	+46.95	+46.78	....	− .20	− .37
XX	+42.12	....	+41.82	+41.74	....	− .30	− .38
XXXX	+46.12	....	+45.75	+45.60	....	− .37	− .52

The **President**. If there are no other papers on sugar, I will call for the report of the referee on tannin.

REPORT ON TANNIN.

By Wm. H. Teas, *Referee.*

For this year's experimental work the following samples were sent to 18 chemists: Chestnut extract, quebracho extract, and a union liquor.

The work on the extracts consisted in a comparison of results on soluble solids obtained by the use of single and double filter paper (S and S 590). Analyses were made according to the official method, the optional use of any good grade of filter paper for nontannin filtration being the only change.

Double and Single Filters.

The question of the comparative influence of different amounts of filter paper in contact with tannin solutions upon the results on soluble solids led to these experiments. It is understood that the filter paper does absorb tanning matter, and this being the case, it is argued that the least amount of paper essential to a clear filtrate should come in contact with the solution. Mr. H. C. Reed undertook some experiments along these lines which are more complete than circumstances will permit reporting in detail. His results as regards filter paper known as 590 S and S were as follows:

Results on soluble solids with single filter for fourteen tanning solutions, ranging from a clear solution of a tanning extract to quebracho solutions, showed an average increase of 0.47 per cent over the results secured on the same solutions by the use of double filters; the official method was used in all cases.

The results of the work on this year's samples show an increase in soluble solids where the single filter was used over the average of double filter results on soluble solids, of 0.23 per cent in the case of the chestnut extract, and an increase of 0.74 per cent on the quebracho extract. This, too, despite the fact that the analyses were made by different chemists and the evaporation and drying took place under different conditions. In general the individual results bear out the indications of the average, there being but two results (both on chestnut extract) in which the soluble solids with double paper exceeded results from single paper. The single filter method requires a longer time for filtration and there is a possible chance of error here, unless all possible precautions against evaporation are taken.

On the question of double or single filter paper, some of the collaborators have expressed themselves as follows:

Mr. Wilson says: "I am convinced that 590 S and S single with assistance will trap all insolubles, and it is certainly desirable to have the least possible amount of paper in contact with a tannin solution. The difference is not so great in quick filtering solutions like chestnut, but on slow filtering solutions, rich in insolubles, the difference is marked, which should, we think, bring the 590 single strongly into favor."

Mr. Reed favors the single paper.

Mr. Alsop is of the opinion that single paper will remove all insolubles.

Mr. Alden says: "I hope the double filter will be retained, for there seems to be a considerable difference in the case of quebracho. The filtrate was not clear, as in the case of the double filter."

Turbid filtrates have caused the referee to hesitate to recomend the adoption of the single paper; with the double paper, kaolin, and ordinary care, a clear quebracho filtrate can always be secured. Single paper has not always given clear filtrates and sometimes necessitates two or three trials. If the single filter gives as clear a filtrate as the double, then there can be no question as to the correctness of the higher soluble solids results obtained from the single filter. It is only with unclarified quebracho and similar extracts that the annoyance of turbid filtrates occurs; and as it is questionable whether the analysis of such extracts represent their true value in the tannery—because of the dissolving temperature, and strength at which they are used differing greatly from the conditions in the laboratory—it is extremely important that every particle of insoluble matter be determined.

Apropos of this matter, I desire to quote from Mr. Alsop's report to the referee: "I regret to say that I did not notice that you said to take 10 grams quebracho to the liter. I took 25.32 grams for 2 liters, which, while it falls within the limits prescribed by the method, will probably make my results low in tannin." This statement indicates a condition of affairs which is patent to every chemist who has had any experience with the analysis and use of extracts rich in insolubles.

With the advantages and disadvantages of the single filter paper in mind, I advocate its trial for determinations of soluble solids, provided that all dryings shall be made from perfectly clear filtrates.

ACID DETERMINATIONS.

Acid determinations by three methods were made on the union liquor sent out. I may here remark that formaldehyde was used on the liquor to keep the initial amount of acid constant. The distillation method directions were as follows:

Take 125 cc of original liquor, distill over 100 cc, and titrate distillate with phenolphthalein and N/10 sodium hydrate. Call this result 80 per cent of total volatile acid in the liquor (i. e., 80 per cent of acid in 100 cc of original liquor).

This method was formerly in use in the laboratories of the United States Leather Company, the procedure of calling the acid in the first 100 cc, 80 per cent of the acid in 100 cc of the liquor having been determined on, after a series of experiments made some years ago. The standard method for comparison used in these experiments I am unable to give, as the data are not now at hand. It has been regarded as a fair comparative method for the volatile acid determination in tan liquors, the low results having been attributed to the percentage of lactic acid in the liquor, which results from this method would not show.

At the suggestion of the referee, Mr. H. C. Reed made some experiments on the determination of acid in acetic and lactic acid solutions of known strength by the three methods indicated by the referee. In these experiments commercial grades of acid were used; 125 cc of acetic and lactic acid solutions and 125 cc of a solution of the two combined, in the ratio of 50 per cent each, were distilled to 100 cc distillate, and the distillates titrated with N/10 sodium hydrate. The following results were obtained:

Acetic acid solution containing 0.664 per cent showed 0.69 per cent.
Lactic acid solution containing 0.58 per cent (as acetic) showed 0.019 per cent.
Acetic and lactic (equal parts) containing 0.623 per cent (as acetic) showed 0.371 per cent.

Experiments conducted on the same lines, but with chemically pure acids, gave the referee the following results:

Acetic solution containing 0.84 per cent showed 0.44 per cent.
Lactic solution containing 0.88 per cent showed 0.011 per cent.
Acetic and lactic solution containing 0.86 per cent showed 0.22 per cent.

Acid determinations made by any distillation method can not include the lactic acid content, as it can not be distilled unchanged, but is broken up by heat into lactic acid, and ultimately into lactide. Distillation methods involving the distilling over of nearly all the original liquor subject the acid to the danger of decomposition by the greater heat of the parts of the still not covered by the residue.

Of the distillation methods used in this year's experiments, Mr. Wilson writes: "The chief objection to the distillation method is in assuming the 100 cc of distillate to contain only 80 per cent of the total acid in the liquor, although the results so obtained seem to bear out the assumption."

Determinations by the Kohnstein-Simand method were made by heating 100 cc of the original tan liquor with 3 to 4 grams of freshly ignited chemically pure magnesia, filtering, removing lime, and determining gravimetrically the magnesia in the filtrate. Theoretically this is probably the best method, but it is too tedious and requires too much care and time to be of practical use in a laboratory where many acid determinations are made. The filtration of the liquor is slow, and, in the case of many heavy liquors from acid hemlock tanneries, it is impossible unless the funnel is kept hot by means of a hot-water jacket.

Mr. Reed reports: "Determinations were made (Kohnstein-Simand method) in triplicate upon solutions of acetic and lactic acids and a combination of the two, in the ratio of 50 per cent each, with the following results:

"Acetic acid solution 0.663 per cent by titration showed 0.664 per cent.

"Lactic acid solution 0.536 per cent by titration showed 0.581 per cent.

"Acetic and lactic acid solution 0.579 per cent by titration showed 0.623 per cent.

"The method does not permit of very rapid determinations, and even if it gives more accurate results, which is doubtful, the writer believes it should be discarded for this reason."

Mr. Wilson says: "The magnesia method might be, when properly handled, the most accurate of the methods under consideration. It has the objection, however, of being tedious, and is open to sources of error in manipulation."

For the charcoal methods 100 cc of the original liquor were diluted to 500 cc and the diluted solution was used for the methods (*a*) and (*b*):

(*a*) To 100 cc of the diluted liquor add 2½ grains of animal charcoal, stir thoroughly every fifteen minutes for two hours, throw on filter and titrate an aliquot portion of the clear filtrate with N/10 sodium hydrate, after the addition of about 200 cc of distilled water.

(*b*) To 100 cc of diluted liquor add 2½ grains charcoal, bring to boiling point with constant stirring, filter, and titrate.

For comparable results by the charcoal methods it is essential that the animal charcoal shall be free from acids and carbonates; the charcoal sent out with the samples is an imported product and has given even results for some six months past.

Results by these methods are generally lower than the actual acid content, but the difference with every lot of charcoal of the best grade is constant, and if a correction for this difference be made the result is nearly absolute.

The objection to method (*a*) seems to be to the length of time the charcoal is in contact with the liquor; results from method (*a*) are invariably lower than those obtained from method (*b*). The difference, however, is not due entirely to the time of contact, but to some extent to the decreased volume of liquor caused by evaporation while heating. Mr. Reed reports on his experiments with acid solutions with these methods as follows:

Two samples of charcoal were used in these experiments, one furnished by the referee and the other an impure grade purified by treatment with hydrochloric acid and washing. Methods (*a*) and (*b*) were used, the latter modified by using a flask with tube condenser and frequent shaking. Charcoal from referee is marked (*c*) and that prepared by writer (*d*):

TABLE I.—*Comparative results obtained with two charcoals.*

Volume of solution.	Acetic acid solution.		Lactic acid solution.		Combination (50 per cent acetic, 50 per cent lactic).	
	(c)	(d)	(c)	(d)	(c)	(d)
	cc	*cc*	*cc*	*cc*	*cc*	*cc*
40 cc solution required without treatment N/10 NaOH	8.85	8.85	7.75	7.75	8.30	8.30
40 cc solution required by method (*a*) N/10 NaOH	6.95	7.70	5.3	7.55	5.90	7.65
40 cc solution required by method (*b*) N/10 NaOH	7.1	7.95	5.35	7.65	5.95	7.80

It will be noted that charcoal purified by the writer gave almost theoretical results in the case of the lactic acid solution, but with the acetic acid solution considerable loss of acid resulted. By treatment of the acetic acid solution with various amounts of a charcoal and calculation of the loss of acid from a given weight of charcoal, practically theoretical results were obtained.

The referee's experiment along the same lines with the same charcoal as that sent out resulted as follows:

TABLE II.—*Comparison of two charcoal methods.*

Volume of solution.	Acetic acid.	Lactic acid.	Acetic and lactic.
	cc	*cc*	*cc*
40 cc solution required without treatment, N/10 NaOH	10.8	10.2	11.4
40 cc solution required by method (*a*) N/10 NaOH	9.4	8.8	9.9
	9.4	8.8	9.8
	9.35	8.8	9.9
	9.4	....	....
40 cc solution required by method (*b*) N/10 NaOH	9.9	9.15	10.1
	9.9	....	....

The use of purified bone black was suggested in the instructions in making determinations by this method, the process consisting of treatment with dilute hydrochloric acid, subsequent washing and drying, and a final heating almost to the point of combustion. Mr. Reed's results with charcoal treated in this manner are given above. The referee heated some charcoal similar to that accompanying the samples and on the liquor sample made the following determinations: By method (*a*), 0.40 and 0.40 per cent; by method (*b*), 0.42 and 0.415 per cent.

From the table given on the determination of acid by the three methods, the Kohnstein-Simand method seems to give the least concordant results. As stated above, the slow filtration and the number of distinct operations necessary seem to afford too many opportunities for error.

The distillation method as used in this work depends too much on the 80 per cent hypothesis. In this case the results are close to those from the charcoal methods, but it has been the referee's experience that many liquors showing a certain percentage of acid by the distillation method will give almost double when treated with charcoal.

It seems to be the opinion of the majority of the collaborators that the charcoal methods are sufficiently accurate and comparable for adoption. The following comments were made on this point:

Mr. Wilson.—It seems to the writer that the simple charcoal method is sufficiently accurate for the control of yard liquors.

Mr. Reed.—The writer is inclined to the charcoal methods for the determination of total acids, preferring method (*b*) with the variation of a flask and tube condenser.

Mr. Small, differing from these views, writes: "The methods outlined (Kohnstein-Simand excepted) introduce such large errors in the amount of acid contained, by reason of the treatment necessary to get clear liquors, that they are valueless save for comparison." He advocates the acid method in which the tannin is precipitated by gelatin and the resulting liquid titrated, using spot test with litmus for end reaction.

TABLE III.—*Results of experiments with chestnut extract, using single S. and S. 590 filter paper.*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
F. P. Veitch	....	43.48	42.93	0.55	17.92	25.01
J. H. Yocum	56.15	43.84	43.52	.33	19.22	24.30
F. H. Small	56.40	43.36	42.40	.96	17.43	24.97
C. H. Brown	55.61	43.94	43.23	.71	17.20	26.03
W. K. Alsop	....	43.72	43.18	.54	17.46	25.72
Frederick Alden	....	43.35	42.80	.55	18.42	24.38
H. T. Wilson	56.17	43.48	[a]42.51	1.02	....	25.78
	56.15	43.53	42.34	1.19	16.73	25.61
H. C. Reed	....	43.76	43.18	.58	17.57	25.61
C. C. Hildebrand	....	43.33	42.88	.45	17.23	25.65
W. H. Teas	....	43.43	42.43	1.00	16.92	25.51
		43.32	42.36	.96	16.89	25.47

[a] Laboratory paper.

	Per cent.
Average soluble solids	42.81
Lowest soluble solids	42.34
Highest soluble solids	43.52
Greatest difference from average	.71

TABLE IV.—*Results of experiments with chestnut extract, using double S. and S. 590 filter paper.*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
F. P. Veitch	....	43.48	42.62	0.86	17.92	24.70
W. H. Krug	....	43.04	41.52	1.52	15.80	[a]25.72
J. H. Yocum	56.15	43.85	43.74	.12	19.22	24.51
F. H. Small	56.40	43.32	42.29	1.03	17.49	24.80
C. H. Brown	55.61	43.94	43.00	.94	17.20	25.80
W. K. Alsop	....	43.72	43.07	.65	17.46	25.61
Frederick Alden	....	43.51	43.03	.48	18.60	24.43
H. T. Wilson	56.17	43.48	[b]42.26	1.22	16.73	25.53
	56.15	43.53	42.33	1.15	16.73	25.60
H. C. Reed	....	43.76	42.91	.85	17.57	25.34
C. C. Hildebrand	....	43.33	42.25	1.08	17.23	25.02
W. H. Teas	....	43.43	42.32	1.11	16.92	25.40
		43.32	42.21	1.11	16.89	25.32

[a] Dried five hours in vacuo, at 100° C.

[b] Laboratory paper.

	Per cent.
Average soluble solids	42.58
Lowest soluble solids	41.52
Highest soluble solids	43.74
Greatest difference from average	1.16

TABLE V.—*Results of experiments with quebracho extract, using single S. and S. 590 filter paper.*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
H. T. Wilson	52.49	....	[a]43.02	4.52	....	35.98
	52.44	47.54	43.08	4.46	7.04	36.04
F. P. Veitch	....	48.00	43.68	4.32	7.07	36.61
J. H. Yocum	52.39	47.61	43.28	4.33	9.06	34.22
F. H. Small	52.42	47.10	42.72	4.38	7.25	35.47
C. H. Brown	52.22	47.69	43.28	4.41	7.42	35.86
W. K. Alsop	....	47.41	42.46	4.95	7.56	[b]34.90
Frederick Alden	....	48.03	45.35	2.68	7.39	37.96
H. C. Reed	52.50	47.47	43.39	4.08	7.13	36.26
Harry Gerber	52.13	48.72	46.23	2.49	7.31	[c]38.92
C. C. Hildebrand	....	47.68	43.54	4.14	7.81	35.78
W. H. Teas	....	47.46	44.32	3.14	7.43	36.89
		47.45	44.46	2.99	7.47	36.99

[a] Laboratory paper. [b] Twelve and one-half grams extract to liter. [c] Fifty cc evaporation.

	Per cent.
Average soluble solids	43.75
Lowest soluble solids	42.46
Highest soluble solids	46.23
Greatest difference from average	2.48

TABLE VI.—*Results of experiments with quebracho extract, using double S. and S. 590 filter paper.*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Non-tannins.	Tannins.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
H. T. Wilson	52.49	....	[a]42.61	4.93	....	35.57
	52.44	47.54	42.23	5.31	7.04	35.19
H. C. Reed	52.50	47.47	42.93	4.54	7.13	35.80
F. P. Veitch	....	48.00	43.40	4.60	7.07	36.33
W. H. Krug	....	48.23	42.20	6.03	6.93	[b]35.27
J. H. Yocum	52.39	47.61	43.00	4.61	9.06	33.94
F. H. Small	52.42	47.09	42.39	4.70	7.30	35.09
C. H. Brown	52.22	47.69	42.83	4.86	7.42	35.41
W. K. Alsop	....	47.41	41.72	5.69	7.56	[c]34.16
Frederick Alden	....	47.75	43.64	4.11	7.17	35.47
Harry Gerber	52.13	48.72	46.05	2.67	7.31	[d]38.74
C. C. Hildebrand	....	47.68	43.16	4.52	7.81	35.35
W. H. Teas	....	47.46	43.02	4.44	7.43	35.59
		47.45	42.97	4.48	7.47	35.50

[a] Laboratory paper.

[b] Dried 5 hours in vacuo at 100° C.

[c] Twelve and one-half grams to liter.

[d] Fifty cc evaporation.

	Per cent.
Average soluble solids	43.01
Lowest soluble solids	41.72
Highest soluble solids	46.05
Greatest difference from average	3.04

TABLE VII.—*Comparison of results on soluble solids.*

Results compared.	Chestnut.		Quebracho.	
	Single paper.	Double paper.	Single paper.	Double paper.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Average soluble solids	42.8	42.58	43.75	43.01
Lowest soluble solids	42.34	41.52	42.46	41.72
Highest soluble solids	43.52	43.74	46.23	46.05
Greatest difference from average	.71	1.16	2.48	3.04

TABLE VIII.—*Results with different methods of determining total acidity.*

Analyst.	Distillation.	Kohnstein-Simand.	Charcoal.	
			(a)	(b)
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
H. T. Wilson	0.355	0.350	0.397 .393	0.426 .420
H. C. Reed	.3945	.4458	.42	[a].4725
F. P. Veitch	.38	.60	.41	.45
J. H. Yocum	.40	.44	.34	.35
F. H. Small	.36	.65	.40	.42
C. H. Brown	.37	....	.37	.48
W. H. Alsop	.37	.48	.36	.42
Frederick Alden	.438	....	[b].44	....
Harry Gerber	.37	.67	.39 .368	.44 .4425
C. C. Hildebrand	.352 .354	....	.375 .38	.45 .409
W. H. Teas	.360	.53	.38	.416
J. H. Norton	.31	.73	.39	.46

[a] When condenser was used, the result was 0.42 per cent; with purified boneblack it was 0.4125.

[b] Three hours' contact.

RECOMMENDATIONS.

As the official method for total acid in tan liquors for this year, the referee proposes the following:

To 100 cc of diluted liquor in a flask with tube condenser add 2 grams chemically pure animal charcoal, bring to boil with frequent shaking, filter, cool, and titrate an aliquot portion.

The following provisional method is also proposed:

To 100 cc of diluted liquor add 2½ grams animal charcoal, stir every fifteen minutes for two hours, filter, take an aliquot part and titrate.

It is convenient to take 40 cc of the filtrate and titrate with N/10 sodium hydrate, as this amount is soon filtered and admits of rapid calculation.

It is highly imperative that the charcoal be free from acid and carbonates and preferable that it be recently freshened by careful heating. Treatment of impure charcoals with dilute acid, followed by ample washing and then heating, will result in a charcoal which will give good results.

The following modifications of the official method are recommended:

To omit the moisture determination.

To change (*b*) in paragraph III so as to read:

Evaporations shall take place under precisely the same conditions as to contact with steam or with a metallic plate. All dryings called for after evaporation shall be done by one of the following methods, under precisely the same conditions, so that the different residues of each analysis may occupy the same shelves in the drying oven:

1. For eight hours at the temperature of boiling water in steam bath.
2. For six hours at 100° C. in air bath.
3. For five hours in vacuo at 100° C.

V. SOLUBLE SOLIDS, to read as follows:

Single pleated filter paper (S. and S. 590, 51 cm.) shall be used. To 2 grams kaolin add 75 cc of the tannin solution, stir, let stand fifteen minutes and decant as much as possible (not on the filter); add 75 cc of the solution, stir, and pour on the filter; keep filter full, reject the first 150 cc of filtrate, evaporate and dry next 100 cc. The portion dried for determination shall be perfectly clear and evaporation during filtration must be guarded against.

VI. NONTANNINS: Two grams kaolin to be used when filtering.

Omit paragraph VIII on testing hide powder, and section (*b*) of paragraph IX.

For next year's work the referee recommends additional experimental work on soluble solids, acid determinations, and on the amount of extract taken for analysis.

The PRESIDENT. I understand that there is one more paper to be presented on tannin, but as Mr. Frear, chairman of the committee on food standards, is compelled to leave to-night, we will listen to his report now, if there is no objection.

REPORT OF THE COMMITTEE ON FOOD STANDARDS.

By WM. FREAR, *Chairman*.

MR. PRESIDENT AND GENTLEMEN: Your committee on food standards would respectfully report with reference to the work of formulation of definitions and standards for foods as follows:

The association has reason for congratulation upon the fact that the conservatism, earnestness, and efficiency of its work, which have won pleasant recognition of its public usefulness in the past, are given distinct recognition by Congress in the appropriation act of the last session for the Department of Agriculture. This act states that "The Secretary of Agriculture, in collaboration with the Association of Official Agricultural Chemists and such other experts as he may desire to consult, shall fix standards for all food products for the information and guidance of the officials and courts charged with the execution of pure-food laws."

During the past year a number of sets of definitions and standards have been revised and prepared for submission to the trade, members of the association charged with food control work, State dairy and food commissioners, and others, to serve as a basis for suggestion and criticism that may assist the committee in perfecting these definitions and standards so as to make them representative of the legitimate trade of to-day. For assistance in the distribution of these schedules the committee is under special obligations to the honorable Secretary of Agriculture and the chief of the Bureau of Chemistry.

Your committee has considered the suggestions from the trade, and has practically completed its work upon two schedules; and it hopes to have ready for submission by the end of the year sets of definitions and standards for the following principal classes of goods: Meat, milk, cereal products, salad oils, spices, wines, vinegars, malted liquors, tea, coffee, and cocoa products, relative to most of which correspondence is now in progress with the trade interests concerned.

In view of the authorization previously granted to this committee, enabling it to bring its work at given stages to completion during intervals between the meetings of the association, and of the desirability of accompanying the definitions and standards, when issued, with a full statement of the principles governing the committee in their formulation, it has been thought preferable not to present at this date the definitions and standards for the several schedules upon which the work of the committee has been practically completed.

It is respectfully requested that the committee be continued.

Mr. WILEY. I have just received a telegram from Dr. Smith, director of the Agricultural Experiment Station of Michigan, stating that Dr. Kedzie is seriously ill and his recovery is possible, but improbable.

On motion, the secretary was instructed to send a telegram of sympathy on behalf of the association.

[The following telegram was sent in accordance with these instructions: "The association sends cordial sympathy to Dr. Kedzie and earnest hopes for a speedy recovery."

Dr. Kedzie lingered until November 7, when his death took place at his home at Agricultural College. The following brief biography and the portrait which constitutes the frontispiece of this bulletin are given place as the last tribute of the association to the memory of one who had long been a valued and beloved coworker.—The Editor.]

A MEMOIR OF THE LATE R. C. KEDZIE.

By L. S. MUNSON.

Robert Clark Kedzie was born at Delhi, N. Y., January 28, 1823. At the age of 3 years he went with his parents to Michigan, where they settled in Lenawee County. They were among the pioneers of that section, and the father having died soon after settling in that region, it was left to the mother and seven children to make for themselves a home under these adverse circumstances. At the age of 17 Robert entered Oberlin College, and graduated six years later. Meanwhile he had supported himself largely by work outside of college duties and by teaching school during vacations. Immediately upon graduation he took charge of Rochester Academy, in Oakland County, which position he held for two years. He then entered the medical school of the University of Michigan, and in 1851 received the degree of M. D. with the first graduating class of that institution. After practicing medicine first at Kalamazoo for one year, and later at Vermontville for ten years, he enlisted with the Twelfth Michigan Infantry. He returned to Michigan in 1863, and was at once appointed professor of chemistry at the Michigan Agricultural College. This position he held until July 1, 1902, when he was relieved of active duties at his own request, and was made professor emeritus.

In 1863 Oberlin College bestowed upon Dr. Kedzie the degree of M. A. In 1898 the Michigan Agricultural College, in recognition of his long and valued service as a scientist, honored him with the degree of D. Sc., and in 1901 the University of Michigan conferred upon him the highest honor in its power, the degree of LL. D.

It was in his capacity as professor of chemistry at the Michigan Agricultural College that his work stands out most prominently. He became connected with that institution at a time when its fate was hanging in the balance; an agricultural college was a new institution and had to demonstrate its usefulness before the people of the State. While Dr. Kedzie had in his own mind no doubt as to the value of such an educational institution, the demonstration of its usefulness was a matter of extreme difficulty. In many cases sentiment was against the college. The legislature was hostile, and support was obtained with difficulty. It was against these odds that the authorities had to work, and to no other man is more credit due for the ultimate success of the institution than to Dr. Kedzie. Not only in its formative period, but in all its subsequent history, he was a dominating factor. A persistent worker, an untiring fighter when once he felt that he was in the right, and a man with conscientious regard for the welfare of the common people, he made his influence felt, not only at the college and throughout the State, but through these institutions he became a man of national reputation.

During his service of thirty-nine years at the college he gave instruction in chemistry to every class save two. He was a natural teacher, and had the faculty to a marked degree of infusing into his students the enthusiasm for his subject that he himself felt. There was no place in his class room for the idler, but for the student he always manifested a keen interest. His classes were always the most popular at the college. The work of the students was always of the first order, not because he compelled it, but because respect for the man commanded it.

In characterizing his services to science and to mankind, Dr. Victor C. Vaughn, of the University of Michigan, speaks as follows:

> I know of no other man who has done so much for the betterment of human life. He was the first to investigate the dangers of arsenical wall papers and to inaugurate legislation looking to the discontinuance of their manufacture. He also investigated the dangers of inferior illuminating oils, and legislation safeguarding the people against them was enacted. Dr. Kedzie has justly been called the father of the beet-sugar industry in Michigan. He showed that something could be grown on the lands where the pine forests had been cut off. As a member of the State board of sanitation he was a leader. He accomplished things. The State of Louisiana, through his efforts, inaugurated a quarantine at New Orleans, the first of its kind in the world, and when Asiatic cholera appeared at the port of New York it was largely because of his influence that it was possible to say: "Thus far shalt thou come, and no farther."

Dr. Kedzie was one of the original members of the Michigan State board of health, and acted as its president for four years. He served as chemist of the experiment station from the time of its establishment in Michigan until his death, and it was largely through his efforts that the inspection of commercial fertilizers was established in the State. Twenty-five years ago he fought and won the battle for winter wheat that the millers were determined to grade below quality. He took an active interest in the work of the Association of Official Agricultural Chemists, and in 1898 he was elected president of the association.

Personally Dr. Kedzie was an exceptional man. His strong, vigorous mind, his frankness of manner, his forceful presentation of facts, his sympathetic and loving spirit won for him the kindest regard and the respect of all who knew him. He had those qualities that make a great man. He had to a remarkable degree the power of concentrating his mind upon a subject until he had completely mastered the facts; and he had the courage to assert what he believed, regardless of whose interests it affected. In the class room and out, the aim to get at the truth asserted itself and made its impression upon those with whom he came in contact. No student ever had a firmer friend than was Dr. Kedzie; no deserving student ever went to him in trouble without receiving some form of assistance.

Dr. Kedzie died November 7, 1902, after an exceedingly distressing illness of about six weeks. He was taken with an affection of the heart which made breathing difficult, yet, through all his suffering, the same invincible will power, the same heroic tenacity of purpose bore him up. Those who knew him personally feel his death as a great personal loss, and the loss of his influence will be severely felt by all who have been associated with him and especially by the institution with which he was so long connected.

Mr. WILEY. I move that the discussion of the report on food standards as well as the reports and discussion on tannin be postponed until to-morrow as we must now give up this room.

The motion prevailed.

On motion by Mr. Davidson the association adjourned at 4 p. m. until 9.30 Friday morning.

SECOND DAY.

FRIDAY—MORNING SESSION.

The meeting was called to order by the president at 9.30 a. m.

The PRESIDENT. The first report in order is that on the separation of nitrogenous bodies, by Mr. Van Slyke.

REPORT ON THE SEPARATION OF NITROGENOUS BODIES.

By L. L. VAN SLYKE, *Referee.*

The referee decided to confine his work this year to the proteolytic products of milk and cheese in the hope of preparing methods of estimation which could be later subjected to critical examination at the hands of different analysts. The work covered by this report has been largely done in the laboratory of the New York Agricultural Experiment Station, at Geneva, by Mr. E. B. Hart. This report is more accurately entitled "Methods for the estimation of the proteolytic compounds contained in cheese and milk."

When milk-casein and its salts, or paracasein in cheese and its salts, are acted upon by dilute acids or alkalies under certain conditions, or by enzyms, or by micro-organisms, the proteid bodies are split up, forming a variety of complex cleavage products. The extent and intensity of such proteolytic changes are measured by the proportions and kinds of the different compounds resulting from the decomposition. In the ripening process that takes place in cheese, we have an instance of extensive proteolysis in the case of the nitrogen compounds of the fresh cheese, due probably to the combined action of dilute acid, enzyms, and micro-organisms. In our study of the causes that produce cheese-ripening and of the conditions that influence the process, we were, at the start, brought face to face with the difficulty involved in the lack of satisfactory methods for making quantitative separations and determinations of the products of cleavage formed. While we have well-elaborated methods for estimating the amount of casein and albumin in fresh milk, these are of little aid in studying the products of their decomposition. Little attention has been given to the development of methods that are applicable to the products occurring in this field. Serious difficulties are involved in such a study. First, we know at present only in an extremely meager and vague way the chemical constitution of milk-casein and and its allied compounds. As regards the constitution of the various compounds formed by the proteolysis of casein, we are largely in the dark, except in the case of some of the simpler ones. In the second place, this field is difficult to study, because the quantities of individual compounds that we have to work with are usually very small. Under these conditions, methods of quantitative separation must, at best, be regarded as largely empirical and more or less tentative. Instead of estimating individual compounds, about which our chemical knowledge is complete, we are compelled, in a large degree, to estimate groups of compounds, the individual members of which we know, for the most part, incompletely or not at all.

From the peptic digestion of casein, Chittenden has separated the proto- and deutero-caseoses,[a] and from a tryptic digestion, a deutero-caseose and a peptone.[b]

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[a] Studies in Physiol. Chem., Yale Univ., 2: 156 (1884–85).

[b] Studies in Physiol. Chem., Yale Univ., 3: 66 (1887–88).

Alexander[a] has separated hetero-caseose from a peptic digestion of casein, but only in small quantities. Many investigators have observed paranuclein or pseudonuclein, the insoluble residue remaining from the peptic digestion of casein or paracasein. Emil Fischer and P. A. Lavene[b] have obtained pyrrolidin-α-carbonic acid from a tryptic digestion of casein.

By hydrolysis of casein with hydrochloric acid, Cohn[c] obtained 1-parahydroxyphenyl-α-amidopropionic acid (tyrosin), 1-α-amido-isobutylacetic acid (leucin), amidosuccinic (aspartic) acid, α-amidoglutaric (glutamic) acid, a pyridin derivative, and ammonia; by the same means, Emil Fischer[d] has recently isolated, among the monoamido acids, in addition to products previously obtained by others, amidovaleric acid, phenyl-α-amidopropionic acid (phenylalanine), pyrrolidin-α-carbonic acid, and probably amido-acetic acid (glycocoll).

Among the crystallizable end products which have been found in ripening cheese are the following: Tyrosin, leucin, histidin, α-ε-diamidocaproic acid (lysin), tetramethylenediamine (putrescin), pentamethylenediamine (cadaverin), lysatin, guanidin probably, and ammonia. Most of these products have been recently reported by Winterstein and Thöny[e] as being found in Emmenthaler cheese. Excepting cadaverin and guanidin, they had also been previously found, though not reported, in American cheddar cheese in this laboratory. It is very probable that other amido compounds will be found, sooner or later, among the products formed by the tryptic, if not the peptic, digestion of milk-casein and of cheddar cheese.

In cheese we find, earlier or later, during the ripening process, a series of compounds and groups of compounds, which, so far as we know at present, appear in something like the following order of succession: (1) Paracasein, (2) unsaturated paracasein lactate, (3) paranuclein (pseudonuclein), (4) caseoses (albumoses), (5) peptones, (6) amido-acid compounds, and (7) ammonia. After the early stages of ripening we have present at the same time all these different compounds and groups.

We shall consider methods for the separation and estimation of the proteolytic products found, first in cheese, and second in milk, following the general order indicated above. It may be well to say at the outset that in dealing with the separation of nitrogenous bodies so complex in composition as those mentioned above and occurring in very variable quantities, we can hope at present in the study of milk and cheese problems only to approximate accurate quantitative results. While we have in the Nencki method a very accurate means of estimating ammonia, the methods used in separating peptones from amido compounds can not be relied upon to give us more than approximate results.

I. METHODS FOR THE SEPARATION AND ESTIMATION OF THE NITROGEN COMPOUNDS OF CHEESE.

The description and discussion of the methods used for the separation and estimation of the nitrogen compounds of cheese will be presented in the following order:

1. Obtaining sample.
2. Determination of total nitrogen in cheese.
3. Extraction of water-soluble products.
4. Determination of total water-soluble nitrogen.
5. Determination of nitrogen in the form of paranuclein.

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[a] Zeit. f. Physiol. Chem., **25**: 411 (1898).

[b] Zeit. f. Physiol. Chem., **33**: 170 (1901).

[c] Ber. deut. Chem. Ges., **29**: 1785 (1896).

[d] Zeit. f. Physiol. Chem., **33**: 151 (1901).

[e] Zeit. f. Physiol. Chem., **36**: 28 (1902).

6. Determination of nitrogen in the form of proteids coagulable by heat in neutral solution.
7. Determination of nitrogen in the form of caseoses (albumoses).
8. Determination of nitrogen in the form of amido-acid compounds.
9. Determination of nitrogen in the form of peptones.
10. Determination of nitrogen in the form of ammonia.
11. Determination of nitrogen in the form of unsaturated paracasein lactate.

1. OBTAINING SAMPLE OF CHEESE.

A sample of cheese is obtained for analysis by means of a cheese-trier, which enables one to secure a round plug of cheese about half an inch in diameter and four to six inches long. Four or five plugs are drawn, one within a short distance of the center of the cheese, one about an inch from the outer circumference, and the others at points equidistant between the two previous ones taken. Samples thus taken represent practically all different conditions existing in the cheese. After each plug of cheese is removed, about an inch of the end having the rind is cut off, and the rest is placed in a well-stoppered, large-mouthed sample bottle. The end with the rind is dipped once or twice in melted paraffin and then carefully replaced in the cheese, being pushed in a little below the surface. After all the plugs have been taken and the ends properly replaced in the cheese, some of the melted paraffin is poured over the surface to fill up and surround the depressions made by replacing the ends of the plugs. This treatment generally insures the exclusion of molds and prevents abnormal loss of moisture in the portions of cheese near the holes left by the removal of the cheese plugs. This is a matter of much importance, when one intends to keep the same cheese for one or two years for systematic examination.

When all the plugs of cheese needed have been taken, the analysis should not be long delayed. The cheese in the bottle is cut into small pieces with a spatula and stirred within the bottle, in order to mix the whole into as homogeneous a mass as possible.

2. DETERMINATION OF TOTAL NITROGEN IN CHEESE.

We weigh out 1 or 2 grams of the cheese, prepared as described above, for the determination of total nitrogen and treat it according to the Kjeldahl-Gunning method, modified as follows: When the solution has become partially digested, we add a piece of copper sulphate about as large as an ordinary pea. Unless this is done, it will take a long time to convert the organic nitrogen completely into ammonia.

3. EXTRACTION OF WATER-SOLUBLE PRODUCTS.

In a porcelain mortar, we thoroughly mix 25 grams of our cheese sample, prepared as indicated above, with about an equal bulk of clean quartz sand. This mixture is transferred to a 450-cc Erlenmeyer flask, to which we add about 100 cc of distilled water at a temperature of 50° C. The flask is then placed on a water bath or in some place where it can be kept at a temperature of 50 to 55° C., and is allowed to stand for half an hour, being vigorously shaken from time to time. The liquid portion is then decanted through a filter of absorbent cotton into a 500-cc flask. The residue is again treated with 100 cc of water, heated, agitated, and the liquid decanted as before. This process is repeated until the filtrate, after being cooled to room temperature, amounts to 500 cc, exclusive of the fat, which usually is present at the top of the liquid.

The cotton filter mentioned is made of two layers of absorbent cotton prepared as follows: In a glass funnel, we place some absorbent cotton to the depth of about 1 inch; moisten this with water, in order to compact it, and then above this place another layer of cotton of the same thickness. Upon this we pour our portions of cheese extract. This kind of filter allows rapid filtration without the aid of a pump, and is as effective in every way as paper, which requires half a day or more for complete filtration of 500 cc of extract. Several samples of cheese can be extracted at the same time. The upper layer of cotton holds all solid particles and can be returned to the flask for extraction with salt solution.

The method of making a water extract of cheese, as described above, insures the complete removal of all water-soluble nitrogen compounds present in the cheese without danger of coagulating any soluble proteids. The use of water at room temperatures is not, in our experience, equally effective in making a complete extraction of the water-soluble products. Under some conditions, as in the early stages of ripening cheese at low temperatures, small amounts of a body are extracted by water which is precipitated by heat in neutral solution. We are unable to say at present whether this body consists of acid salts of paracasein or of hetero-caseose, which are practically insoluble in water, or whether it is some other compound. The temperature of 50° C. also has the advantage of arresting further peptic or tryptic action during the extraction. The use of acids in extracting cheese is to be avoided, since a small amount of acid will not only precipitate the soluble nuclein, but may form salts with paracasein, which are somewhat soluble in a slightly acid solution; the amount of dissolved paracasein salts under such circumstances depends on the amount of acid used and the time of extraction.

4. DETERMINATION OF TOTAL WATER-SOLUBLE NITROGEN.

For the determination of the amount of total water-soluble nitrogen, we take 50 cc of the water extract, prepared above, equivalent to 2.5 grams of cheese, and treat it according to the Kjeldahl method for determining nitrogen.

5. DETERMINATION OF NITROGEN IN THE FORM OF PARANUCLEIN (PSEUDONUCLEIN).

To 100 cc of the water extract, equivalent to 5 grams of cheese, we add 5 cc of a 1 per cent solution of hydrochloric acid, and warm the mixture on the water bath at 50 to 55° C. until complete separation takes place, as shown by a clear supernatant liquid. The precipitate is filtered, washed with water, and then, with the filter paper, treated by the Kjeldahl method to determine the amount of nitrogen. The nitrogen equals nitrogen present in the form of paranuclein (pseudonuclein.)

In our early work 2 or 3 cc of a saturated alum solution were used for this determination, for the reason that in the separation of casein in milk we had used this reagent successfully; but at the time the nature of the body we were precipitating from our water solution of cheese was not known. Later, when we had studied it and learned its character it was found, on comparing precipitations, by use of alum and by hydrochloric acid that alum gave high results, undoubtedly precipitating some caseoses. In 27 comparative trials with water extracts of different cheeses, we found in the alum precipitate nitrogen varying from 0.2 to 0.337 per cent of the cheese and averaging 0.269 per cent, while the nitrogen in the hydrochloric acid precipitate varied from 0.046 to 0.145 per cent of the cheese and averaged 0.085 per cent. The nitrogen precipitated by alum in these 27 cases was from 2.1 to 5.5 times as much as that precipitated by hydrochloric acid, the average of all being 3.2. Since hydrochloric acid is known to precipitate paranuclein completely, we are justified in assuming that the alum precipitates other compounds, and this is confirmed by other work, showing that when alum is used as the first precipitant we get smaller quantities of caseoses in the filtrate than we do when we use hydrochloric acid as the precipitant of paranuclein. Alum appears to resemble zinc sulphate as a precipitant of proteids.

Paranuclein (pseudonuclein) results from the breaking down of casein or paracasein and is always found in the water extracts of ripening cheese, whether salted or unsalted. It may, perhaps, be regarded more accurately as a residue and probably should not be counted as one of the products to be used in measuring the extent of cheese ripening. This is undoubtedly the same body as Chittenden's dyspeptone,[a] which he found as an insoluble residue in a pepsin-hydrochloric-acid digestion of casein.

6. DETERMINATION OF NITROGEN IN THE FORM OF PROTEIDS COAGULABLE BY HEAT IN NEUTRAL SOLUTION.

The filtrate from the preceding determination (5) is made neutral with dilute caustic potash, using phenolphthalein as an indicator. It is then heated at the temperature of boiling water, until any coagulum that forms settles completely, leaving a clear supernatant liquid. The precipitate is washed with water and its nitrogen determined by the Kjeldahl method. In our experience, such a precipitate rarely occurs, except in the case of cheese ripened near freezing point. The nature of this body we have not yet investigated.

7. DETERMINATION OF NITROGEN IN THE FORM OF CASEOSES (ALBUMOSES).

The filtrate from the preceding determination (6) is treated with 1 cc of 50 per cent sulphuric acid, saturated with chemically pure zinc sulphate and then warmed to about 70° C. until the caseoses separate completely and settle. The mixture is allowed to cool and is then filtered. If filtered hot, there will occur a further separation of caseoses in the filtrate on cooling. The precipitate is washed with a saturated solution of zinc sulphate made slightly acid with sulphuric acid. The nitrogen in the precipitate is determined by the Kjeldahl method.

For the determination of caseoses, the use of ammonium sulphate was exclusively employed until Bömer[b] proposed the use of zinc sulphate, which possesses a distinct advantage in enabling one to determine nitrogen directly in the precipitate or filtrate. This method has been employed, in the estimation of caseoses, also by the Wisconsin Agricultural Experiment Station. In the present state of our knowledge of this class of compounds, zinc sulphate must be regarded as the most available reagent for their quantitative separation.

8. DETERMINATION OF NITROGEN IN THE FORM OF AMIDO-ACID COMPOUNDS.

The amido-acid compounds are determined in the filtrate from the precipitation of peptones (9). For the removal of peptones three reagents have been commonly used: (1) Tannin and sodium chlorid, (2) phosphotungstic acid with sulphuric acid, and (3) bromin with hydrochloric acid. After the removal of peptones the filtrate contains amido-acid and ammonia compounds. After determining the amount of total nitrogen in this filtrate and then the amount of nitrogen present in the form of ammonia (as obtained in 10, p. 97), we subtract the amount of ammonia nitrogen from the combined amount of amido-acid and ammonia nitrogen and thus obtain the amount of amido-acid nitrogen. In the following section (9) we describe the methods involved in removing peptones by the different reagents and the efficiency of each reagent.

9. DETERMINATION OF NITROGEN IN THE FORM OF PEPTONES.

(1) *By tannin and sodium chlorid.*

We place 100 cc of our water extract of cheese in a 250-cc graduated flask, add 1 gram of sodium chlorid and a solution containing 12 per cent of tannin until one drop added to the clear supernatant liquid gives no further precipitate. We then dilute to the 250-cc mark, shake, filter through a dry filter, and determine the amount of nitrogen in 50 cc of the filtrate by the Kjeldahl method; this gives us the amount of nitrogen in the form of amido-acid and ammonia compounds. The amount of nitrogen in the form of peptones is determined by difference—that is, by subtracting from the amount of total nitrogen in the water extract the combined sum of the amounts of nitrogen found in 5, 6, 7, 8, and 10.

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[a] Studies in Physiol. Chem., Yale Univ., 3:66 (1887–88).

[b] Zeit. Analyt. Chem., 5:562 (1895).

The combination of tannin and salt has been settled upon by us as the most satisfactory for the separation of casein-derived peptones from amido-acid compounds in milk and cheese analysis, when, as is commonly the case, we have large amounts of amido-acid compounds relative to peptones. We have confirmed Schjerning's[a] results showing that this reagent does not precipitate the monoamido-acid compounds, such as leucin, tyrosin, aspartic acid, glutamic acid, and amidovaleric acid, nor does it precipitate histidin, arginin, lysin, cadaverin, putrescin, lysatin, or ammonia. In our work the tannin-salt solution has nearly as great a precipitating power as phosphotungstic acid, precipitating 93.3 per cent of the total nitrogen compounds present in a sample of fresh milk; in a study of ripened cheese it precipitated the uncrystallizable end products, caseoses and peptones, so completely that no further trouble was experienced in separating the crystallizable end products.

It is well to record here the fact that when precipitation of peptones with tannin-salt solution is attempted in a mineral acid solution no precipitate occurs; it is only in neutral solution that more complete precipitation takes place.

The chief objection to the use of tannin-salt solution as a means of separating caseoses and peptones from amido-acid compounds and ammonia is that it is not a complete precipitant of peptones. Hence, when the reagent for this separation is used we commonly leave some peptones to be estimated as amido-acid compounds, the amount of peptones thus being made smaller and the amount of amido-acid compounds larger than the quantity actually present. Under the discussion of the use of phosphotungstic acid as a reagent for separating these classes of nitrogen compounds are given for comparison some results secured by each of the two reagents.

(2) *By phosphotungstic acid with sulphuric acid.*

In a 250-cc graduated flask we place 100 cc of the water extract of cheese, add 100 cc of water and then 5 cc of strong sulphuric acid. To this add phosphotungstic acid of 30 per cent strength until one drop gives no further precipitation in the clear supernatant liquid. Then dilute to the 250-cc mark and filter through a dry filter. In 50 cc or 100 cc of this filtrate we determine the amount of nitrogen by the Kjeldahl method, and then the amount of peptones is obtained by difference.

Phosphotungstic acid has come into very general use in this country and in Europe as a means of separating peptones from amido-acid compounds in work with cheese and milk. The results of Stutzer[b] and of Bondzynski[c] agree in showing that phosphotungstic acid is a complete precipitant of casein, caseoses, and peptones, while in their experience it does not precipitate the amido-acid compounds or ammonia. Freudenreich and Jensen, in all their work, even of recent date, have used this reagent in the cold as a means of separating peptones from amido-acid compounds. Babcock, Russell, and Vivian, in this country, have used it, as well as tannin, designating the different precipitates as "peptones by phosphotungstic acid" and "peptones by tannin."

In the introductory portion of this paper, when mentioning different cleavage products of casein, we included among them the hexon bases, viz, arginin, histidin, and lysin; and, in addition, certain compounds resulting from their cleavage, such as putrescin and cadaverin, and also pyrrolidin-α-carbonic acid, all of which are precipitated by phosphotungstic acid. While all these products have not been separated from ripening cheese, it is probable that they will be sooner or later. We shall soon publish results of work done in this laboratory showing in normal ripening cheese the presence of histidin, lysin, putrescin (derived from arginin[a]), and Siegfried's lysatin, which is also precipitable by phosphotungstic acid. The quantities of these bases that can be derived from casein are not to be neglected, since, for example, in a hydrochloric-acid cleavage 15.4 per cent of the nitrogen of the products splits off into the hexon bases. Bondzynski has used phosphotungstic acid in hot solution with arginin, and finds the precipitate soluble when hot, separating out on cooling; and this statement we can confirm, the solution, however, being complete only on boiling. In the case of lysin, histidin, and putrescin the phosphotungstic-acid precipitate fails to redissolve completely at the temperature of the water bath or on boiling. This behavior renders worthless the use of phosphotungstic acid as a reagent for the separation of peptones from those amido-acid compounds that are precipitated by it.

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[a] Zeit. Analyt. Chem., **39**: 545 (1900).

[b] Zeit. f. analyt. Chem., **35**: 1493 (1896).

[c] Landw. Jahrbuch der Schweiz (1894).

We have seen that tannin-salt solution fails to precipitate peptones completely, and that phosphotungstic acid precipitates, in addition to peptones, some amido-acid compounds. When, therefore, we use these two reagents in precipitating solutions that contain both peptones and amido-acid compounds, as in the case of normal-ripening cheese, we should expect to find the amount of nitrogen compounds left in the filtrate less with phosphotungstic acid than with tannin. This is found invariably to be the case. Vivian [b] has published some results obtained with nine different cheeses, which illustrate this point. We give his figures in the table following:

Table I.—*Comparison of phosphotungstic acid and tannin-salt solution in precipitating the nitrogen compounds contained in water extracts of cheese.*

Precipitating agent used.	Numbered samples.									Average.
	1.	2.	3.	4.	5.	6.	7.	8.	9.	
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Phosphotungstic acid	1.08	1.00	0.82	0.63	0.67	1.69	0.95	1.10	1.22	1.02
Tannin-salt solution	1.54	1.26	1.12	.65	1.16	1.87	1.08	1.36	1.42	1.27

In connection with the above results nothing was stated in regard to the history of the cheeses used in the work. We have studied the action of phosphotungstic acid and tannin-salt solution in connection with samples of cheese that were under known well-controlled conditions, thus learning something of the general character of the proteolytic changes taking place. Twenty-five grams of cheese curd were placed in each of several Erlenmeyer flasks, 50 cc of water added, and the contents sterilized by heat. We then added 0.5 gram of concentrated lactic acid, in order to convert the paracasein into unsaturated paracasein lactate. [c] To some of the flasks thus prepared sterilized pepsin was added, and to others sterilized rennet extract. For this purpose the enzyms were prepared as follows: We dissolved 600 milligrams of Parke Davis & Co.'s aseptic pepsin in 25 cc of water, added 0.5 per cent of formalin (containing 40 per cent of formaldehyde), and let the solution stand until bacteriological examinations, made by Mr. H. A. Harding, showed the absence of living organisms. The mixture was then diluted to 100 cc with water, and to each flask containing cheese we added 10 cc of this sterilized-pepsin solution. One thousand parts of the mixture in each flask thus contained one part of pepsin.

In preparing the sterilized rennet solution, 5 cc of Hansen's rennet extract were diluted to 25 cc with water, 0.5 per cent of formalin was added, the completeness of sterilization proved by bacteriological examinations, diluted to 100 cc and 10 cc of this preparation added to each flask containing cheese.

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[a] A. Ellinger, Ber. deut. chem. Ges., **31**: III-3183 (1898).

[b] Ann. Rept. Wis. Exp. Sta. **16**: 171 (1899).

[c] N. Y. Agr. Expt. Sta. **214**: 67 (1902).

The flasks thus prepared were kept at 15.5° C. The analytical results given below were obtained at the end of two and four weeks. In this work, we had only the enzyms of pepsin and of rennet pepsin acting upon our proteid. Under these conditions, especially in the given length of time, we should not expect the formation of any considerable amount of amido-acid compounds precipitable by phosphotungstic acid. The results obtained are given in the following table:

Table II.—*Comparison of phosphotungstic acid and tannin-salt solution in precipitating the nitrogen compounds formed by peptic digestion of cheese.*

Precipitating agent used.	Numbered samples.								
	1.	2.	3.	4.	5.	6.	7.	8.	Average.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Phosphotungstic acid	0.10	0.10	0.150	0.175	0.10	0.08	0.16	0.16	0.123
Tannin-salt solution	.45	.44	.645	.685	.48	.46	.73	.70	.574

In every case except one, the amount of nitrogen not precipitated by tannin-salt solution was more than four times the amount obtained by phosphotungstic acid. No ammonia was present in any case. In view of these widely differing results in relation to the amounts of amido-acid compounds found, the questions may be asked: "Which reagent more nearly represents the amount of amido compounds actually present? Does the tannin-salt reagent fail to precipitate the peptones completely, thus allowing the unprecipitated peptones to be counted among the amido-acid compounds? Or does the amount of nitrogen in the filtrate in case of this reagent really represent amido-acid compounds? Does the phosphotungstic acid precipitate some of the amido compounds simultaneously with the peptones, thus cutting out a part of the amido-acid compounds and counting that part among the peptones? Or is there in the cheese only the small amount of end products indicated by the action of the phosphotungstic acid?"

While we did not by individual isolation determine to what extent amido compounds were present, we are justified in believing that such compounds were not yet present in the cheese in any appreciable degree; and, hence, the figures obtained with phosphotungstic acid are much nearer the truth than those obtained by tannin-salt precipitation. The reasons for this belief are as follows: The amido compounds precipitated by phosphotungstic acid are known to be chiefly the diamido compounds, while the monoamido compounds are precipitated little, if at all. Hence, the compounds found in the filtrate of a phosphotungstic acid precipitation are mainly monoamido compounds. Now let us assume temporarily that the amounts of nitrogen in the tannin-salt filtrate, given in the table above, represent the total amido compounds, free from peptones; then, since the monoamido compounds are represented by the amounts of nitrogen obtained in the phosphotungstic acid filtrate, the difference between the two sets of figures—that is, those obtained by tannin salt solution and those obtained by phosphotungstic acid—represents the amount of diamido compounds. Taking the average of the results given in the table above, the nitrogen of the total amido compounds is 0.574 per cent of the cheese, while the nitrogen of the monoamido compounds is 0.128 per cent of the cheese, thus leaving the difference as the nitrogen of the diamido compounds, equivalent to 0.446 per cent of the cheese. According to these figures, the monoamido compounds constitute about 22 per cent of the entire amount of amido bodies, while the remainder, 78 per cent, represents largely diamido compounds. In this case, the ratio of monoamido to diamido compounds is as 1 to 3.5. Keeping these data in mind, we will call attention to some work done by Hart[a] in studying the cleavage end products formed by the action of hydrochloric acid on casein. He found that the diamido compounds formed less than 20 per cent of the total amido compounds, so that the ratio of monoamido to diamido compounds was as 1 to 0.25 or less; in other words, the monoamido compounds were greatly in excess of the diamido compounds or just the reverse of what we find to be the case in the results embodied in the table above, based on the assumption that the nitrogen in the tannin-salt filtrate represents the total amido compounds and nothing more. The most obvious and rational explanation of this discrepancy observed in the ratio of monoamido to diamido compounds is that it is wrong to assume that the tannin-salt filtrate contains only amido compounds and not any peptones. Withdrawing that assumption, then, and allowing that the nitrogen in the tannin-salt filtrate represents some peptones as well as the amido compounds, how can we tell in this particular case the true amount of amido compounds in the cheese? Unquestionably the results with phosphotungstic acid more nearly represent the truth in regard to the amido compounds, because under the conditions of the experiment we should expect very small amounts of amido compounds, if any; and in this particular case the amounts are so small as practically to indicate the absence of amido bodies altogether. From this it may be seen that it is possible for the tannin-salt reagent to give results that are decidedly misleading.

(3) *By bromin with hydrochloric acid.*

To the filtrate from the zinc sulphate precipitate (7) we add 2 or 3 drops of strong hydrochloric acid and then bromin until the liquid becomes saturated and there remains after vigorous agitation an undissolved globule of bromin, amounting to 0.5 cc to 1 cc. This is allowed to stand over night. The precipitate is then filtered and washed with bromin-saturated water. The nitrogen in the precipitate is then determined by the Kjeldahl method and is called nitrogen in the form of peptones, the filtrate containing the amido-acid compounds and ammonia.

The use of chlorin by Rideal and Stewart[b] in precipitating proteids suggested to Allen and Searle[c] the use of bromin. They reported that bromin quantitatively precipitates the products formed by the peptic digestion of egg albumin and they developed the method practically as given above. As applied to the separation of peptones from amido-acid compounds in cheese and milk, the method gives varying results, depending upon the age of the cheese or milk used.

In the case of our water extracts made from cheese nine months to a year old, crystalline bodies in noticeable quantities are precipitated by bromin along with peptones, owing probably to the presence of tyrosin, giving the solution a turbid appearance and rendering filtration difficult. Schjerning[d] has shown that tyrosin behaves in this manner with bromin water. This precipitate is partly retained on the filter paper and is estimated as peptone.

Schjerning has shown also that bromin does not completely precipitate milk proteids and their derived caseoses and peptones. Of the whole proteid he obtained only 76.7 per cent by bromin. In the case of milk we have obtained results varying with the age of the milk. In perfectly fresh milk, when the amount of amido compounds must have been least, we obtained 91.3 per cent of the entire milk proteids by bromin precipitation. In another case of fresh milk we compared the precipitation of proteids by bromin and hydrochloric acid with that by tannin and sodium chlorid and by solution of phosphotungstic acid and sulphuric acid, with the following results: There was precipitated—

By phosphotungstic and sulphuric acids, 93.8 per cent of the total nitrogen.

By tannin and sodium chlorid, 93.3 per cent of the total nitrogen.

By bromin and hydrochloric acid, 91.5 per cent of the total nitrogen.

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[a] Zeit. f. Physiol. Chem., **33**:347 (1901).

[b] Analyst, **22**:228 (1897).

[c] Analyst, **22**:259 (1897).

[d] Zeit. f. Anal. Chem., **39**:545 (1900).

There is also a possible source of error in connection with the use of bromin in precipitating peptones when the filtrate from the bromin precipitate is used directly for the determination of amido-acid compounds. We have found in the case of water extracts from cheese over one year old that there is an actual loss of nitrogen when bromin is allowed to stand in contact with the water extract. In the case of one cheese two years old, the cheese extract, consisting of caseoses, peptones, and amido-acid compounds, contained nitrogen equivalent to 2.74 per cent of the cheese before adding bromin, while, after standing one hour in contact with bromin in hydrochloric-acid solution, there remained only 1.52 per cent of nitrogen; in other words, there had disappeared 44.6 per cent of the nitrogen present before the addition of bromin. In cheese one year old we have found the loss varying from nothing in one case to over 5 per cent in others. To show whether or not this loss came from the action of bromin on the caseoses or peptones we removed the caseoses with zinc sulphate, and in another sample of cheese extract we removed the caseoses and peptones with phosphotungstic acid, and the loss still occurred. By passing a current of air through the above extract in contact with bromin and then through potassium hydroxid and through sulphuric acid, these reagents were found free from nitrogen compounds, indicating that the lost nitrogen disappeared in the form of free nitrogen and not in the form of ammonia or nitrogen oxids. We can not regard the method of determining the amount of peptones in cheese extracts by means of bromin as a reliable method, because (1) bromin precipitates small amounts of tyrosin and perhaps certain other similar compounds; (2) it is not a complete precipitant of caseoses and peptones; and (3) its filtrate can not be used for the determination of amido-acid compounds, especially in old cheeses, owing to the decomposing effect of bromin upon such compounds, setting nitrogen free. In addition, bromin is a most disagreeable reagent to handle.

(*4.*) *Comparative value of different reagents used in separating peptones and amido-acid compounds.*

We have now considered in some detail each of the three reagents most commonly used in separating peptones from amido-acid compounds, viz: (1) Tannin-salt solution, (2) phosphotungstic acid with sulphuric acid, and (3) bromin with hydrochloric acid. Tannin-salt solution fails as a perfect reagent for the separation, because it does not completely precipitate peptones, which results in making the quantitative results for amido-acid compounds too high and may indicate the presence of considerable quantities of amido compounds even when they are practically absent. Phosphotungstic acid, on the other hand, completely precipitates peptones, but also precipitates some of the amido-acid compounds that are present in cheese and milk, and the consequence is that the amount of amido-acid compounds found is too low. Bromin is open to both objections—it fails to precipitate peptones completely and at the same time does precipitate some of the amido-acid compounds. While these two sources of error might tend to offset each other under certain conditions, we can not depend upon such a method for reliable quantitative results.

In our judgment, it is desirable, for best results, to use phosphotungstic acid to separate peptones and amido-acid compounds when the amount of amido-acid compounds is relatively small as compared with peptones or when they consist mostly of monoamido compounds. This condition occurs in the early stages of cheese ripening and persists longer in cheese cured at low temperatures; it occurs also in milk and cheese acted upon by pepsin enzyms, especially in the presence of chloroform.

Tannin-salt solution can be relied upon to give better results than phosphotungstic acid when amido-acid compounds are present in proportions that are relatively large compared with peptones or when they consist largely of diamido compounds. The former condition prevails in normal cheese cured under usual conditions, especially after the first few weeks of curing.

10. DETERMINATION OF NITROGEN IN THE FORM OF AMMONIA.

Distill with magnesium oxid 100 cc of the filtrate from the tannin-salt precipitation, passing the distillate into a standardized acid and titrating in the usual way. In our early work the cheese mass itself, suspended in water, was used for distillation, giving slightly higher results than the method just described. The small increase is generally accounted for as coming from the proteids themselves present in the solution. Theoretically it is true that when such bases are present as putrescin and cadaverin they might distill with the ammonia. In one case, where a large quantity of cheese was subjected directly to distillation with magnesium oxid and the distillate examined for these bases, none was found, the distillate consisting entirely of the ammonia salt. The high boiling points of cadaverin and putrescin and the consequent difficulty of distilling them with steam probably accounts for their absence in the distillate.

In our early work on the determination of ammonia in milk and cheese we subjected to distillation with magnesium oxid and with barium carbonate many different amido compounds in order to ascertain if any of these bodies when pure could split off basic nitrogen. While some of the products used in our work are not at all likely to be found in cheese or milk, we include them with the others in the list given below. The method was carried out as follows: Dissolve 1 gram of each amido body, or if insoluble, suspend it in 50 cc of water, and for distillation use 10 cc of this mixture diluted to 150 cc, adding magnesium oxid or barium carbonate and using ordinary atmospheric pressure.

Amido bodies used were—

Acetamid.
Allantoin.[a]
Arginin.
Aspartic acid.
Creatin.
Creatinin.
Diphenylamin.
Glutamic acid.
Glycocoll.
Histidin.
Leucin.
Lysin.
Phenylenediamin.
Trimethylamin.
Tyrosin.
Uric acid.
Xanthin.

The result of this work was that nothing was distilled over with either magnesium oxid or barium carbonate in any case except that of trimethylamin, the nitrogen of which was all obtained in the distillate with both regents.

We have also used the excellent Nencki apparatus, distilling under reduced pressure the filtrate from the tannin-salt precipitation. In comparative trials we have obtained no lower results than when we distill under ordinary atmospheric pressure.

11. DETERMINATION OF NITROGEN IN THE FORM OF UNSATURATED PARACASEIN LACTATE.

The residue insoluble in water is treated with several portions of a 5 per cent solution of sodium chlorid, the process being carried out as in preparing the water extract (3, p. 89). The nitrogen in an aliquot part of the 500 cc of this salt extract is determined by the Kjeldahl method.

II. Methods for the Separation and Estimation of the Nitrogen Compounds of Milk and their Proteolytic Products.

We will briefly describe the methods used for the separation and estimation of the nitrogen compounds of milk and their proteolytic products in the following order:

1. Determination of total nitrogen in milk.
2. Determination of nitrogen in the form of casein.
3. Determination of nitrogen in the form of albumin and syntonin.
4. Determination of nitrogen in the form of caseoses.
5. Determination of nitrogen in the form of amido-acid compounds.
6. Determination of nitrogen in the form of peptones.
7. Determination of nitrogen in the form of ammonia.

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[a] Zeit. f. Physiol. Chem. **34**: 145 (1901).

1. DETERMINATION OF TOTAL NITROGEN IN MILK.

Weigh about 5 grams of milk and determine the nitrogen by the Kjeldahl method.

2. DETERMINATION OF CASEIN.

To about 10 grams of milk add 90 cc of water at 104 to 108° F. (40 to 42° C.) and then 1.5 cc of 10 per cent acetic acid. Agitate and warm at the temperature given above until a flocculent precipitate separates, leaving a clear supernatant liquid. Filter, wash, and treat by the Kjeldahl method for estimating nitrogen.

In fresh milk, 2 or 3 cc of a saturated solution of alum may be used in place of acetic acid, usually with little higher results. But when the milk casein has been proteolyzed to any extent, the use of alum is not permissible, since it precipitates caseoses in addition to casein.

The use of acetic or any other acid in precipitating casein in milk, whose casein has been digested in any degree, precipitates, in addition to casein, any paranuclein that is present. We have not yet succeeded in devising satisfactory methods for the separation of these two compounds.

3. DETERMINATION OF NITROGEN IN THE FORM OF ALBUMIN AND SYNTONIN.

The filtrate from 2 is neutralized by caustic alkali, using phenolphthalein as indicator, and is then heated at the temperature of boiling water until the precipitate completely separates and settles. The precipitate is then filtered, washed, and treated by the Kjeldahl method.

4. DETERMINATION OF NITROGEN IN THE FORM OF CASEOSES.

The filtrate from 3 is heated to 70° C., 1 cc of 50 per cent sulphuric acid is added and then chemically pure zinc sulphate to saturation. Let stand at the temperature indicated until the caseoses completely separate and settle. Then cool the mixture, filter, wash with a saturated solution of zinc sulphate made slightly acid with sulphuric acid, and treat the precipitate by the Kjeldahl method.

5. DETERMINATION OF NITROGEN IN THE FORM OF AMIDO-ACID COMPOUNDS.

Treat about 50 grams of milk with a tannin-salt solution or with phosphotungstic acid according to directions given under 8, p. 91.

6. DETERMINATION OF NITROGEN IN THE FORM OF PEPTONES.

From the total nitrogen, subtract that found in all forms other than that of peptones, as indicated under 9, p. 91.

7. DETERMINATION OF NITROGEN IN THE FORM OF AMMONIA.

See under 10, p. 97.

III. DETERMINATION OF CHLOROFORM.

When chloroform is used as an antiseptic in milk and cheese it is very essential to know approximately the amount present, in order that we may have a proper control of conditions. We have used the following method successfully: Place 5 grams of milk or cheese in a pressure bottle with about 100 cc of alcohol and 5 grams of caustic potash. The bottle is then heated in an autoclave for half an hour at 110° C. The resulting chlorid is determined by titration, as for chlorin in sodium chlorid.

RECOMMENDATION.

It is recommended that the methods presented above be adopted as provisional methods under the head of dairy products.

Mr. Bigelow. I would like to ask if the referee has tried precipitating with bromin in the presence of zinc sulphate. Some time ago we examined meat extracts by dividing the filtrate from the proteoses (coagulated by neutralization and boiling) into several portions, and precipitated one portion with bromin and another with zinc sulphate. It was expected that bromin would precipitate both proteoses and peptones, whereas zinc sulphate would not precipitate peptones. Mr. Trescot, who was doing this work, was very much surprised to find that the zinc sulphate precipitate was greater than the bromin precipitate.

The President. If there is no objection on the part of the association I would like to introduce the president of the Columbian University.

ADDRESS BY PRESIDENT NEEDHAM COLUMBIAN UNIVERSITY.

Mr. President and Gentlemen of the Association: It would be the height of presumption for me to undertake to discuss any of the matters which this distinguished body of chemists is considering. I belong to another profession, and in these days one profession is quite enough for one man. I come to extend my congratulations upon this large and representative gathering of your association; to welcome you on behalf of Columbian University to this building, and to offer you all the accommodations and facilities at our disposal for your meetings. We are honored and delighted to have scientific men come here and discuss in public meetings the progress and results of their work; it is one of the great features of Washington life and is due in a great measure to the fact that we have so many eminent scientific men here engaged in professional work. The number employed by the Government is little known. I was much interested in a statement made to me by an official at the time Mr. Carnegie made his noble gift to found in this city an institution for original research. I said to this gentleman, "What a great sum of money Mr. Carnegie has given to scientific work." And he replied, "Yes, ten millions is a large sum, but do you know what the Government of the United States is doing for science?" I replied that I did not. And then he told me that the amount of money expended annually by the Government for scientific work represented an annual interest of 2½ per cent upon $200,000,000.

I have been desirous to secure the services of some eminent men, who are in larger and older institutions, for this university, and feared that they could not be induced to come for the reason that they would have to leave the association and intellectual atmosphere of the older universities, but I was pleased to be told by a most distinguished educator that this would not prevent their coming—"Because," he said, "you have so many distinguished scientific men in the Departments of the Government among whom they would find association in the city if it were not found to a sufficient degree in the university."

A few years ago I was crossing the Atlantic, homeward bound, and saw among the passengers three very distinguished looking men. They were always together, and much absorbed in earnest conversation. I overheard them speaking of their "profession" with great respect and veneration. I thought they must be clergymen. I learned that two of them were from a German and one from an English university, and their "profession" was chemistry. It was then something new for me to hear chemistry spoken of in such exalted terms as a profession. It impressed me, and set me to thinking. I recalled that the greatest service, and, therefore, the greatest nobility, lies along the path of discovering the elements and forces of nature; the way in which these forces may be made serviceable to man; in short, discovering the laws of nature. It has been said that the great object of education should be to acquire the knowledge of some one thing, and hold it with such a grip as to make it of practical value to mankind. We are passing away from the old thought that only the mysterious is divine, to the broader and better thought that an understanding of the laws of nature is also a revelation of the divine thought. There can be no higher or nobler calling than that in which men, enthusiastic with a desire to know the truth, are seeking and unfolding a knowledge of the uses and laws of matter. When we look about and undertake to enumerate the things which science is doing for the good of man, we must be profoundly impressed with the fact that if these results could be withdrawn the world would be deprived of its richest treasures. No greater service has been rendered to this great land in the development of its almost boundless natural resources than by the scientists of America. They have purified the foods that nourish us; they have multiplied and cheapened the products of the mines and the factories; made the fields yield larger and richer harvests; and made the desert "to blossom as the rose."

I welcome you here, and congratulate you upon the dignity of your labors, the nobility of your calling, and the splendid achievements of your profession.

The PRESIDENT. I am sure that I express the feeling of every member of the association in thanking Mr. Needham most cordially for his appreciative and inspiriting words.

If there are no other papers to be read on the separation of nitrogenous compounds we will proceed to the discussion of the subject.

Mr. VAN SLYKE. In discussing the matter from the standpoint of the paper read this morning it should be remembered that while some general methods may be applied to the nitrogen compounds obtained from different sources, as from meat and vegetables, we can not expect that one man can go to work and apply the same method in a wholesale way to each of these different groups of compounds, until we know something more in detail concerning the individual compounds and their chemistry. So, while from personal reasons, and on account of the character of the investigation we are carrying on we have given attention exclusively to this side of it, we do not mean to imply that these methods have a general application. The same kind of work must be done for each class of compounds, and we are liable to fall short in our appreciation of the enormous amount of work involved and of the great number of compounds with which we have to deal. I do not offer this as an apology for the work which we have done, but simply as an explanation of the paper presented and the large amount of work which remains to be accomplished.

The PRESIDENT. I am sure every member of the association appreciates the character of the work which Mr. Van Slyke and Mr. Hart have done, and we know that one man can not be expected to cover the field. It might be possible to associate the best experts in the country with the work in connection with meat extracts and cheese and vegetable products. This would seem to be a matter worthy of consideration.

If there is no further discussion on this question, we will hear the report on soils.

REPORT ON SOILS.

By F. P. Veitch, *Referee.*

Your referee did not know until the receipt of Dr. Wiley's notification in March that he had to serve in this capacity. Upon receipt of this notice, samples were solicited and the following circular letter requesting cooperation and outlining the work was sent to the chemists of all the stations and of the U. S. Department of Agriculture. Only eight promised to cooperate in any part of the work.

Outline of Proposed Work on Soil Samples for 1902.

Dear Sir: Below you will find an outline of the proposed work on soils for this year. The work is divided into three parts, one or more of which you are asked to take up, notifying the referee of your selection.

(1) The determination of available phosphoric acid.
(2) The determination of available potash.
(3) The analyses of alkali soils and crusts.

The samples for the first two lines of work have been selected very carefully from the permanent fertilizer plats of several of the experiment stations and represent soils differing greatly in physical as well as chemical conditions.

The methods with which it seems most desirable to work are indicated under each heading. At the same time the referee would be pleased to have comparative results by other methods, for which, of course, full credit will be given.

As it is not probable that one worker will be able to assist in all the work outlined, each is asked to select the line or lines which he feels he can undertake, and notify the referee, who will forward appropriate samples.

It is hoped that three or four careful workers will make determinations of available plant food by the N/200 HCl method and that others will make determinations by the N/5 HCl method.

The referee would urge upon the members of the association the desirablity of a more hearty cooperation in the study of methods for soil investigations, and to this end he would suggest to those holding professorships in their respective State colleges the suitability of this line of investigation for a portion of the work required for the Master's degree.

Determination of moisture is to be made by the official method, Bureau of Chemistry, Bulletin No. 46, revised, page 71. Calculate all results to a free-water basis.

1. *Determination of available phosphoric acid.*

(It is asked that in work on this subject preference be given the N/200 HCl method.)

(*a*) By the N/200 HCl method (Jour. Am. Chem. Soc., **24**, p. 79).

(1) Follow the method strictly as given, except do not correct for basicity of sample.

(2) Uniform temperature (40° C.) and agitation must be secured. Use Wagner machine.

(3) Results should be corrected by blanks used on reagents.

(*b*) By the N/5 HCl method (Bulletin No. 46, revised, p. 74).

(1) Uniform temperature (40° C.) and agitation must be secured. Use Wagner machine.

2. *Determination of available potash.*

(*b*) By the N/200 HCl method.
(1) Prepare solution as for the determination of phosphoric acid.
(2) Results should be corrected by blanks on reagents.
(*c*) By the N/5 HCl method.
(1) Use a portion of the solution secured for phosphoric acid determination.
(2) Correct results by using blank on reagents.

3. *Analysis of alkali soils and crusts.*

By direction of the association the methods of alkali analyses as carried out in the Bureau of Soils, United States Department of Agriculture, were referred to the referee for study and report.

For this work no samples will be furnished by the referee, but those interested in this line of work are requested to give these methods a trial and to suggest such modifications as experience shows will improve them. For these methods see Bulletin No. 18, Division of Soils, page 65.

As the result of some preliminary study the referee would suggest, for the consideration of those interested, the following modifications of the methods:

(*a*) Under "Preparation of solution," page 67, in place of treating 50 grams of soil with a liter of water in the large beaker, the referee suggests that 10 grams be placed in a liter flask, or 5 grams in a half-liter flask, made up to volume, shaken frequently, and allowed to stand overnight.

(*b*) For standard solution of acid potassium sulphate use N/10 sulphuric acid. (North and Lee find that strictly theoretical results are obtained when the point of the burette is below the surface of the liquid being titrated; also that the presence of about one-half gram sodium chlorid in the volume titrated introduces no appreciable error.—Jour. Soc. Chem. Ind., No. 5, XXI, p. 322, 1901.)

(*c*) As indicators in dark solutions, compare methyl orange and Congo red.

(*d*) On page 70, line 26, for "several drops of a solution of phenolphthalein" read "1 cc of a 1 per cent solution of phenolphthalein."

(*e*) On page 72, line 26, for "one drop" read "three drops."

(*f*) As the estimation of the bases begins with lime, a word of caution should be given. Before adding ammonium oxalate, add ammonia in slight excess; and if a precipitate forms, boil, filter, and proceed as usual. This is necessary in soils or crusts containing large amounts of carbonates, which, of course, dissolve some alumina.

All results must be in the hands of the referee by October 1, 1902.

Please notify the referee promptly whether or not you can cooperate in this work.

Yours, very truly,

F. P. Veitch, *Referee on Soils.*

U. S. Department of Agriculture, Bureau of Soils,
Washington, D. C., May 1, 1902.

The Samples.

In soliciting samples for the work the referee has aimed to get soils whose crop and fertilizer history is known accurately for a number of years, and which will be representative of different kinds of soils. In view of the fact that so few cooperate in this work, it has also appeared desirable so to select such samples that soil from these same plats may be used in the future for the association work, if it appears desirable. The referee believes that more will be accomplished by testing various methods on a series of known soils than by trying them on new samples each year, as it will then be possible to compare the results of many workers by several methods on known soils.

The history of the plats before the experiments were started is given in the text under the respective sample numbers. The crop history since the beginning of the experiment now being conducted on these plats is summarized in Table III. In all cases where it is possible the referee has calculated the effect of an essential plant food by subtracting the yield of the plats to which all but this particular plant food was applied from the yields of the plats receiving complete fertilizers.

The referee desires to acknowledge the assistance of the directors and chemists of the several stations, who not only furnished the samples, but in the majority of instances distributed the subsamples to the cooperators.

DIRECTIONS FOR TAKING SOIL SAMPLES.

All samples should be taken from a sufficient number of places in a plat to be thoroughly representative of the plat. The number of places should never be less than two and may be more, being governed by the size and uniformity of the plat.

In taking samples the undecomposed matter on the surface is to be avoided as far as possible.

Samples should be taken to the full depth of the soil; that is, to the change in color, texture, or other conditions usually recognized as marking the line between soil and subsoil, provided this line does not occur at a greater depth than 1 foot. In no case is a sample to be taken to a greater depth than 1 foot.

The depth to which samples are taken should be noted and a statement of the depth should accompany the sample.

Samples should not be taken when the soil is too wet to work.

Sufficient soil should be taken to give about 100 pounds of dry soil which will pass through a 3-mm mesh sieve.

Samples should be dried as quickly as possible at a temperature not over 40° C.

In those cases where the subsamples are distributed directly by the collector, the samples when dry should be passed through a 3-mm sieve, breaking down all lumps with a wooden mallet or rolling pin, the fine and coarse material weighed, and the fine material wrapped in strong paper in packages of about 1 pound.

Cotton sacks and franks will be furnished by the referee for the distribution of the samples.

In addition the referee should be furnished with as full a description as possible of the obvious characteristics of the subsoil, drainage conditions, distance from surface of solid rock when this distance is less than 10 feet, together with such other data as may be of value.

SAMPLE NO. 1.

This was taken from the nitrogen plats of the five-year rotation experiments at Wooster, Ohio. The soil is a yellow somewhat sandy clay, lying upon the upper rocks of the Waverly series. It is of glacial origin, but is largely modified by the soft sandy shales upon which it lies, and which have been ground up and mixed with material derived from granite and limestone to the northward. The native rock is abundantly streaked with iron, and a liberal percentage of iron is found in the soil.

The original forest growth of this region was chiefly white oak, with a little admixture of red, scarlet, and black oaks, and an occasional chestnut. The most striking arboreal feature is the thick undergrowth of dogwood.

The topography of the country is rolling, owing entirely to erosion, as the rocks lie in level strata. When the forest was cleared away, the slowly decaying roots of the deep-rooted white oak furnished channels of drainage through the thin sheet of clay to the loosely stratified rocks below, and through their rifts and seams the drainage waters percolated to feed the multitude of springs for which the region is famous. But as the roots decayed more completely the plow and the tramping of teams and pastured stock obliterated these natural channels and artificial drainage became necessary.

The following tables exhibit the results of analyses of this soil:[a]

TABLE I.—*Mechanical analysis of soil from Wooster, Ohio.*

Constituent.	Size of particles.	First 6 inches.
	Millimeters.	*Per cent.*
Gravel	2 to 1	0.56
Coarse sand	1 to 0.5	.86
Medium sand	0.5 to 0.25	.71
Fine sand	0.25 to 0.1	1.79
Very fine sand	0.1 to 0.05	20.47
Silt	0.05 to 0.01	29.97
Fine silt	0.01 to 0.005	36.07
Clay	0.005 to 0.0001	4.74

[a] Ohio Agr. Expt. Sta. Bul. 110.

TABLE II.—*Chemical analysis (1.115 HCl) of soil from Wooster, Ohio.*

Constituent.	First 6 inches.	Second 6 inches.	Third 6 inches.
	Per cent.	*Per cent.*	*Per cent.*
Insoluble	88.71	87.64	88.17
Potassium oxid (K_2O)	.22	.28	.25
Sodium oxid (Na_2O)	.39	.38	.39
Calcium oxid (CaO)	.32	.31	.31
Magnesium oxid (MgO)	.36	.35	.36
Ferric oxid (Fe_2O_3)	2.64	3.29	2.97
Aluminum oxid (Al_2O_3)	2.53	3.49	3.01
Phosphoric acid (P_2O_5)	.80	.80	.80
Sulphuric acid (SO_3)	.04	.05	.05
Loss on ignition	4.48	4.08	4.25

The experiments on this land were commenced in 1888. Dissolved boneblack and dissolved rock have been used as the standard carriers of phosphoric acid; muriate of potash as the source of potash; and nitrate of soda furnished the standard for nitrogen. Where phosphates have been applied it has been at the rate per acre of 320 pounds; muriate of potash at the rate of 260 pounds; nitrate of soda, 440 pounds; and dried blood, 40 pounds, during a five-year rotation.

Incidentally it may be said that nitrogen or potash alone or together have not been profitable, and where ammonium sulphate is the carrier of nitrogen the yields have been smaller than with sodium nitrate.

SAMPLE NO. 2.

This sample is from plat No. 6 (muriate of potash and dried blood plat) of the rotation experiments of the Pennsylvania experiment station. The soil is a magnesium limestone clay loam, about 8 inches in depth, through which fragments of flint are scattered. It has good natural drainage and contains a fair supply of organic matter. The surface soil is fairly uniform in depth and fertility, but the subsoil varies greatly in depth.

The fertilizers are applied twice during the rotation—to the corn and to the wheat—at the rate of 218 and 250 pounds dried blood, 250 pounds dissolved boneblack, and 200 pounds muriate of potash per acre.

Applications of lime have reduced the yields decidedly, while sulphate of ammonia has given greater yields than nitrate of soda.

SAMPLES NOS. 3 AND 4.

These samples are from plats Nos. 10 and 11 of the phosphoric acid experiments at the Maryland station. The soil is a rather stiff clay, naturally quite well drained, though quite level. In general character the plats are quite uniform, in fact more so than most pieces of like area in this formation. The history of the cropping of the land used for this test was, so far as known, as follows: There was a poor stand of grass and weeds on this land in 1888, which was plowed down and seeded to wheat, which was harvested in 1889; grass, 1890–91; corn, 1892; fallowed 1893; wheat, 1894. Clover and timothy were sown in the wheat and gave a good stand. It was limed at the rate of 30 bushels of stone lime per acre in 1893. Since the beginning of the experiments in 1895, muriate of potash and nitrate of soda have been applied in like amounts to all plats, while the source of the phosphoric acid has been varied. Sample No. 3 has not received phosphates since 1880, while sample No. 4 has received in five applications, ground raw S. C. rock at the rate of a total of 2,650 pounds per acre.

SAMPLE NO. 5.

Sample No. 5, from Amherst, Mass., is furnished by the Massachusetts agricultural experiment station from plat O, field F; a friable loam, with a retentive subsoil of clay that is nearly filled with gravel and small stones in the lower strata. Previous to 1887 the field had been used as a meadow and was well worn-out, at that time yielding but a scanty crop of English hay.

The plat (No. 5) in proximity to the one from which the sample was taken, and with which comparisons will be made later, received the following treatment: In 1887 the sod was turned under. The crops grown in 1888, and in succession since, have been Hungarian grass, forage crops, potatoes, wheat, serradella, maize, barley, rye, soy beans, Swedish turnips, and maize. The fertilization began in 1890 with about 500 pounds of dissolved boneblack, 290 pounds of nitrate of soda, and 390 pounds of potash-magnesium sulphate. This application was made yearly till 1895, after which time no more phosphoric acid was added, but the yearly addition of the other ingredients was increased one-half. In 1893 and thereafter the same crops were grown upon the plat (No. 0) from which the sample of soil was secured as upon the above-mentioned one, but the first addition of fertilizer was made in 1896, since which the application of nitrogen and potash has corresponded with that on the other plat. So far as is known, no phosphate has ever been added to plat 0. Both plats were limed at the rate of a ton of quicklime per acre in the spring of 1898. It will be seen that, during 1896, 1897, and 1898, the fertilization of the two plats was the same, but previous to these years plat 0 had received no fertilizer, while plat 5, during a series of years, had received a total application of about 1,900 pounds of nitrate of soda per acre, 2,500 pounds of potash-magnesium sulphate, and 2,500 pounds of dissolved boneblack.

SAMPLE NO. 6.

Sample No. 6 is from plat No. 1 wheat acre of the Kentucky station. Soil is derived from the Lower Silurian limestone, rich in phosphoric acid. The top soil is a brown clayey loam; the subsoil, a yellow clay so retentive as to cause the land to be deficient in natural drainage; the surface soil, 8 to 12 inches deep. It has been in wheat continuously since 1887 without fertilizer of any kind. The soil of the plat does not now produce a crop of wheat. It is of the same character as that of the corn acre, and the data given in regard to it will apply as well to the soil of the corn acre.

SAMPLE NO. 7.

This sample is from plat No. 4 (nitrogen and phosphoric acid plat) of the "soil-test" experiment of the Rhode Island station.

The soil here mentioned is from the "plain." It is of a dark chocolate color, about 4 inches deep, underlaid by an impure clay resting upon gravel and pebbles of unknown depth, which form a good natural drainage. The soil is evidently derived from gneiss, consisting of quartz, orthoclase, and biotite, and made up of glacial drift.

Mechanical analysis.[a]

	Per cent.		Per cent.
Moisture in air-dried sample	1.90	Fine sand, 0.25–0.1 mm	6.66
Organic matter	8.46	Very fine sand, 0.1–0.05 mm	31.65
Gravel, 2–1 mm	1.82	Silt, 0.05–0.01 mm	29.69
Coarse sand, 1–0.05 mm	3.40	Fine silt, 0.01–0.005 mm	4.38
Medium sand, 0.5–0.25 mm	7.82	Clay, 0.005–0.0001 mm	4.81

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[a] Rhode Island Agr. Expt. Sta. Bul. 28.

Chemical analysis of air-dried fine soil.[a]

	Per cent.		Per cent.
Insoluble matter	80.632	P_2O_5	0.127
K_2O	.155	Moisture and organic matter	7.888
CaO	.569	Humus	2.752
MgO	.209	Ash from humus	.605
Fe_2O_3	3.463	P_2O_0 in humous ash	.042
Al_2O_3	4.641		

It was found[b] that the amount of phosphoric acid extracted from these soils, rich in organic matter, was increased 41 per cent by substituting the Goss for the above method.

Soil taken from different portions of the same field in 1895 contained, on a water-free basis, humus, 3.86 per cent (by the Huston method, which gives higher results than the Hilgard method, in most cases); humous nitrogen, 0.13 per cent; and free humic acid,[c] 1.52 per cent (Rhode Island station). Soil which had been exhausted of its available potash by fertilization with nitrogen and phosphoric acid, taken from the same field in 1897, yielded only 0.020 per cent of lime by treatment with carbonated water.[d] The soils under discussion give an acid reaction to litmus paper, and fail to produce satisfactory yields in the case of many crops without an addition of lime or other alkaline materials to correct the existing acidity.

In 1890 an experiment was begun with maize to indicate the relative amounts of more active nitrogen, phosphoric acid, and potash within the soil. Previous to this time grass had been growing for many years without fertilization, and hardly enough was produced at the time the experiment began to pay for cutting. In 1890 and 1891 nitrate of soda, 150 pounds; dissolved boneblack, 350 pounds; and muriate of potash, 150 pounds per acre were applied. In 1892 and subsequently the annual application was increased to 480, 600, and 200 pounds, respectively. In 1896 air-slacked lime was applied to all plats at the rate of 2½ tons per acre.

A plat to which a complete fertilizer was applied was not included in this experiment until 1899.

SAMPLE NO. 8.

This soil is from plat No. 67 of the limed series, phosphoric acid experiments, Rhode Island station. No phosphoric acid has been added to this plat.

This land is of the same general character as No. 7. In this experiment there are two series of plats on which the comparative value of different phosphates is being studied. One series is limed, the other is unlimed. Each plat, except the check plats, received, in the course of each year from 1895 to 1899, inclusive, like amounts of phosphoric acid, irrespective of the form. All have received like amounts of nitrate of soda and of muriate of potash. Owing to the very acid character of this soil, which vitiates the results obtained with some of the crops raised, it has been thought best to give the increase due to phosphoric acid of slag meal as well as from the standard phosphoric acid carrier, dissolved phosphate rock.

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[a] Rhode Island Agr. Expt. Sta. Bul 28. Collected 1892. Hilgard method.

[b] U. S. Dept. Agr., Division of Chemistry, Bul. 47.

[c] Method given in Wiley's Agricultural Analysis, vol. 1, p. 331.

[d] U. S. Dept. Agr., Division of Chemistry, Bul. 56.

CHARACTER OF SAMPLES AS INDICATED BY CROPS GROWN.

The data compiled in Table III show the increase in the crops obtained by adding plant foods to the various soils of which these samples are representative:

TABLE III.—*Increase in crops per acre due to plant food added to soils from which samples were taken.*

Soil sample No.	Plant food added.	Wheat.		Oats.		Corn.		Hay.	Barley.	
		Grain.	Straw.	Grain.	Straw.	Grain.	Stover.		Grain.	Straw.
		Lbs.	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*
1	K_2O	252	507	147	([b])	75	106	106	....	....
	P_2O_5	571	1,163	387	557	646	162	598	....	....
2	K_2O	264	762	77	163	369	1,732	171	....	....
	P_2O_5	480	1,167	134	165	717	1,629	817	....	....
3	P_2O_5	546	1,411	....	....	478	393	375	....	....
4[a]	P_2O_5	....	....	....	....	....	....	....	....	....
5	P_2O_5	....	....	269	131	3,490	1,360	....	726	1,890
6	K_2O	135	....	....	....	3,609		....	....	....
	P_2O_5	160	....	....	....	([b])	([b])	....	....	....
7	K_2O	....	....	....	....	330	1,320	....	....	....
	P_2O_5	....	....	....	....	([b])	([b])	....	....	....
8	P_2O_5,[c] unlimed	....	....	787	5,112	1,041	2,159	1,601	....	....
	P_2O_3,[c] limed	....	....	575	1,725	295	885	1,117	....	....
	P_2O_3,[d] unlimed	....	....	562	2,437	961	1,853	2,661	....	....
	P_2O_5,[d] limed	....	....	472	2,097	458	891	1,166	....	....

Soil sample No.	Plant food added.	Rye.	Cabbage.	Turnips.	Soy beans.		Potatoes.	Tobacco.	Crimson clover.	Beets.
					Beans.	Straw.				
		Lbs.	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Lbs.*	*Bush.*
5	P_2O_5	1,330	983	574	950	1,360	....	....	....	....
6	K_2O	....	....	....	....	....	5,280	268	....	....
	P_2O_5	....	....	....	....	....	2,100	142	....	....
7	K_2O	....	....	....	....	....	2,814	....	....	....
	P_2O_5	....	....	....	....	....	2,406	....	....	....
8	P_2O_5,[c] unlimed	....	....	320	....	....	11,070	....	4,960	9.5
	P_2O_5, limed	....	....	3,665	....	....	6,900	....	460	70
	P_2O_5,[d] unlimed	....	....	522	....	....	9,900	....	6,840	154
	P_2O_5,[d] limed	....	....	6,335	....	....	5,640	....	820	473

[a] Fertilized with ground raw phosphate rock and well supplied with available P_2O_5.

[b] Larger yields without P_2O_5.

[c] From dissolved South Carolina rock.

[d] From slag meal.

From the preceding figures we see that—

Sample No. 1 is very deficient in available phosphoric acid for hay, wheat, oats, and corn. The increased yield of oats and wheat due to use of potash is about half that due to phosphoric acid; with corn and hay it is practically nothing.

Sample No. 2 is very deficient in available phosphoric acid for corn, wheat, grass, and oats, being about like No. 1 in this particular. It is deficient in available potash for corn and wheat, but practically not deficient for grass and oats.

Sample No. 3 is about as deficient in phosphoric acid as No. 1 for wheat, corn, and hay.

Sample No 4 has had only ground raw phosphatic rock applied to it, but experiments for five years with crops show that larger yields have been obtained with insoluble phosphate than with soluble or reverted; hence sample No. 4 should be higher in available phosphoric acid than No. 3.

Sample No. 5 is decidedly deficient in phosphoric acid for corn, soy beans, barley, rye, cabbage, turnips, and oats. Particularly so for corn, barley, rye, and soy beans

Sample No. 6 has never been found to need phosphates, except for potatoes. It is very deficient in available potash for potatoes and corn, somewhat deficient for tobacco, and but little for wheat.

Sample No. 7 is deficient in available potash for potatoes, less deficient for corn; deficient in phosphoric acid for potatoes, but not for corn.

Sample No. 8. is very deficient in available phosphoric acid for potatoes, crimson clover, corn, hay, oats, beets, and turnips; in all cases, except with turnips, much less deficient when limed.

RESULTS OF WORK ON SAMPLES.

Determination of phosphoric acid was asked for in samples Nos. 1, 2, 3, 4, 5, and 8. Determination of potash was asked for in samples Nos. 6 and 7.

CHEMICAL ANALYSES.

The results reported are given in the following table:

TABLE IV.—*Determinations of potash and phosphoric acid by the N/200 hydrochloric acid method.*

[Parts per million.]

Analyst.	Constituent.	Sample No. 1.	Sample No. 2.	Sample No. 3.	Sample No. 4.	Sample No. 5.	Sample No. 6.	Sample No. 7.	Sample No. 8.
C. C. Moore, Bureau of Chemistry (not corrected for basicity).	Potash	52.0	94.0	136.0	50.0	177.0	16.0	11.0	39.0
	Phosphoric acid	.66	1.3	10.0	6.0	0.66	4.0	1.0	1.0
C. C. Moore, Bureau of Chemistry (corrected for basicity).	Phosphoric acid	.7	1.7	12.0	8.0	.70	4.5	1.0	1.0
F. P. Veitch, Bureau of Soils (not corrected for basicity).	Potash	55.0	111.0	142.0	42.0	175.0	18.0	12.0	42.0
	Phosphoric acid	.6	1.2	11.0	7.0	.5	4.0	2.0	1.6

With regard to phosphoric acid we see that samples Nos. 1, 2, and 8, which are low in available phosphoric acid, show less than 2 parts per million, while No. 3, which is also low, shows from 10 to 11 parts, and No. 7, which should be well supplied with available phosphoric acid, shows only from 1 to 2 parts per million. The results on Nos. 3 and 4 are contrary to the crop history of the soils. The other results are in harmony with their crop history.

With regard to potash, Nos. 6 and 7, which are markedly deficient, show but small amounts dissolved by this method, while No. 1, which is somewhat deficient in available potash, shows 52 to 55 parts per million, more than No. 4 or No. 8, which are presumably well supplied. In view of the fact that the samples represent but a fraction of the feeding depth of plants in these soils, it seems advisable to give the method further trial on samples so taken as to represent approximately the feeding ground of plants.

It seems to the referee that in the use of these methods which remove so little from the soil it is particularly necessary to know the solubility of the plant foods to the full feeding depth of the plants. While the advisability of this was recognized, it was not possible to take the matter up this year.

MECHANICAL ANALYSIS.

In the matter of the advisability of the association's adopting methods for the mechanical analysis of soils, the following self-explanatory correspondence is submitted:

SEPTEMBER 12, 1902.

Prof. MILTON WHITNEY,
Chief of Bureau of Soils, Washington, D. C.

DEAR SIR: Among the questions referred by the Association of Agricultural Chemists to the referee on soils was that of the importance of the mechanical analysis of soils and the advisability of the association providing in its published methods for mechanical as well as for chemical methods for soil analysis. It seems to the referee that you are the one who is best fitted to make a statement covering these points, and he therefore respectfully asks that you write a letter to be submitted over your signature as part of the report on soils, stating to what extent the physical condition of a soil as revealed by its mechanical analysis may be regarded as exerting a measurable or practical effect on the fertility or crop-producing power of that soil.

Your discussion of the advisability of changing the present limits of size for the several separates obtained by the standard mechanical method, and of the closeness with which results can be duplicated by the same and by different workers, will also be appreciated.

Yours, very respectfully,

F. P. VEITCH,
Referee on Soils.

SEPTEMBER 30, 1902.

Mr. F. P. VEITCH,
Referee on Soils, Bureau of Soils:

SIR: Your letter of September 12 has been received, referring to the advisability of the Association of Official Agricultural Chemists providing in its published methods for mechanical as well as for chemical methods for soil analysis. This letter has been given full and careful consideration, and I find it a rather difficult question to deal with, as there are so many contingencies that have to be taken into account, which I realize it is difficult for the analyst to inform himself upon and to consider in the examination of individual samples.

Generally speaking, I believe the mechanical analysis throws an important light upon the character of the soil and the character of the crop to which it is adapted, but, as in the chemical analysis, there are questions of climate, physiography, transportation, and markets, as well as other more subtle differences in the structure of soil which have to be given full consideration in using the results of the mechanical analysis.

To reply first to your last inquiry, I think it very undesirable to have a large number of physical separations, as it is impossible for us to use many of these separations in our estimate of the physical properties or the physical character of the soil. As you know, we have adopted seven separations: (1) Fine gravel, (2) coarse sand, (3) medium sand, (4) fine sand, (5) very fine sand, (6) silt, and (7) clay. I think that these various grades should stand, as these differences can be recognized in the field by our soil experts, and it is quite clearly recognized that these several separations occurring in different proportions in the soil, influence in a more or less marked manner the character of the crop adapted to the land, its relation to drainage and agricultural practice. In the correlation of soils into great classes in this Bureau, we are, however, reducing these separations to four principal groups by combining the fine gravel, coarse sand, and medium sand into what we roughly designate as "coarse grades;" the fine sand and the very fine sand into what we roughly consider "fine sand material;" the silt, which has in the field peculiar properties typically shown in a true loess, and clay, which has certain characteristic properties of plasticity, which, however, may be shown by any of the other groups.

It is useful in comparing, for example, the Cecil clay and the Hagerstown clay, or the Cecil sandy loam, the Cecil loam, and the Cecil clay, to see from this abbreviated statement of the results of the mechanical analysis whether the coarse sand, fine sand, or the silt, or the clay predominates, or what the relation between these different grades bear to each other in the different soils or different groups of soils.

The practice of the Bureau has been to simplify, as much as possible, the broad classification of the soils, and for this purpose 16 general classes have been established, viz, stony loam, gravel, gravelly loam, dune sand, sand, fine sand, sandy loam, fine sandy loam, loam, shale loam, silt loam, clay loam, clay, adobe, meadow, and muck and swamp. We endeavor to indicate in the type name that the soil may be thrown into one or another of these general classes, but each one of these classes may contain any number of different types of soils so long as their individual characters and peculiarities are clearly stated.

Furthermore, we have decided, after full and careful consideration, that these classes shall be based upon the field judgment of the men rather than upon the mechanical analysis. It is quite possible that a soil will show from the mechanical analysis 60 or 70 per cent of silt, which would indicate that it belonged to the loess type, and should be called a silt loam, while the properties in the field show it to be a true clay in being extremely plastic and impervious to water and possessing none of the friable properties of a loess. Again, we may have a soil which shows a low content of clay with a high content of fine sand, having in the field an impervious character, high retentive properties for water, and being more or less plastic, in which case the soil would go under the general class of a clay soil regardless of the fact that the mechanical analysis shows the soil to contain relatively little clay.

While these differences may be perplexing to the person who is classifying the results of the laboratory work, they are nevertheless valuable in pointing out essential properties of the soil, which would otherwise be overlooked. A soil containing 70 per cent of silt, but having the properties in the field of a true clay, is interesting from the standpoint of the soil physicist in indicating certain properties the soil possesses differing markedly from a loess containing 70 per cent of silt, and, furthermore, these differences unquestionably give valuable guides as to the proper treatment of these soils, which would not be deduced alone from the mechanical analysis or from the field examination.

If a soil is described as a sandy soil or a sandy loam and the mechanical analysis shows that it contains not to exceed 6 or 8 per cent of clay, I should have no hesitation in stating that early truck crops could be produced on the soil; but whether it would be an economic venture to develop the industry would depend upon climatic conditions, transportation facilities, and market demands. If the soil contains 10 per cent and over I should say that it would be adapted to a different class of truck crops, particularly to the late varieties of potatoes and to the heavy truck crops, such as cabbage. If the soil contains 15 per cent of clay, I should say that it would be unwise to expect any successful development in early truck crops, although it might be admirably adapted to market gardening which could be carried on within hauling distance of a city or town.

With the knowledge we now possess of the influence of the soil upon the commercial type of tobacco, it should be possible, from the mechanical analysis alone, within certain prescribed limits which mark our different tobacco districts, to determine the character of the leaf that would be produced: In the Northern cigar-tobacco districts, whether a wrapper, binder, or filler could be produced on a soil of any given texture; in the Southern districts, whether a bright tobacco, manufacturing tobacco, or the different types of export tobacco could be produced. But it would be unwise to predict either success or failure from similar analyses of soils taken outside of the present recognized tobacco districts. The commercial type of tobacco is so markedly influenced by the physical character of the soil that, in this particular case, as no doubt with many other crops, the commercial type is dependent upon this character, with little, if any, regard to the actual chemical composition of the soil. The chemical composition of the soil undoubtedly influences the yield and quality of crops under these conditions, but it has little noticeable effect upon the type of tobacco which is grown. In other words, the yield, size of leaf, burn, and grain of the leaf can undoubtedly be modified by fertilization, but so far as my experience goes fertilization can not produce a wrapper leaf on a filler soil, as these types are dependent primarily under given climatic conditions upon the texture and physical properties of the soils. Similar relations could be shown between other crops and the physical character of the soil, but it would hardly be justified in the limits of such a brief communication as is probably expected in reply to your letter.

As the operations of the Bureau of Soils extends over wider areas of the country, resulting in the establishment of recognized types and reports as to the relation of these types to crops, it is believed that a basis will be given for the correlation of soils even outside of the areas which have been surveyed and which will make it imperative for the agricultural chemist to adopt methods of mechanical analysis in conjunction with his chemical work in showing the class of crops adapted to the soil in addition to his chemical work which may show the relation of the soil to the actual yield of crops. I consider it inadvisable to increase the number of separations beyond the seven grades which we now recognize. The actual size or limits of these separations is a relatively unimportant matter. It is necessarily an arbitrary measure. Whether the conventional separation in use by the Bureau is adopted or whether the separations vary according to the square root of 10 or some other mathematical relation is unimportant, except for the fact that this Bureau has unquestionably the largest and most representative collection of soil samples that has ever been made, and has made and published upward of 3,000 analyses covering all of the most important types of soil in this country and many from abroad. Furthermore, the soil survey has been extended to 30 States in this country and is rapidly being extended in an immeasurably greater scope than can be given by any individual or State. It would, therefore, seem advisable for the analyst, in view of the work thus far done, and the enormous extension of this work which will undoubtedly be made possible through the liberal appropriations by Congress, to adopt the same separations as are here used. This would insure a uniformity of results. It would make possible a correlation of samples with areas which have been accurately mapped and carefully investigated by this Bureau, and would, in my judgment, add greatly to the value of the work of the individual analyst in comparison with what could be accomplished by a change in the limits of size of the separation at this time by any individual or by any association.

Careful watch has been kept upon the accuracy of the details of the method as shown by duplicate analyses by the same and different individuals in this Bureau, and it is my judgment that the limit of error is much smaller than the justifiable phases which are recognized by our field men, or than we are able to recognize in their effect upon the crop. In a truck soil containing not to exceed 8 per cent of clay, I can see no advantage in working for results closer than 1 or 2 per cent of clay. A difference of half of 1 per cent has no meaning to me. Samples from different parts of the type may differ much more than this with no apparent difference in their relations to crops, and if duplicate results by different workers come within 1 or 2 per cent of the amount of clay in such a soil, it would be perfectly satisfactory to me. On the other hand, in a clay soil the difference between 30 and 35 per cent of clay is unimportant, as I think we can not recognize such differences in crop relations. Again, 50 per cent of silt with certain physical properties places a soil in the class of loess soils. I would not recognize any essential difference in such a type if duplicate analyses or duplicate samples showed 60 per cent of silt, because, in my judgment, the difference between 50 per cent and 60 per cent of silt would not have so marked an effect upon the crop as to be readily recognized. It would certainly be far less liable to affect the crop than would different methods of cultivation. If there was 70 or 80 per cent of silt, this would be a different matter from the case of a soil containing 50 per cent of silt, such as we have just been considering, and I should expect different crop relations and look for different cultural methods as desirable. While I would not advocate any less care in the mechanical analyses than is now given, I feel satisfied that the methods at present used are accurate far within the limits of variation which may be allowed in any type of soil, and far within the limits of variation of the effect of the soil on the crop. We must not forget that while we want accuracy in our laboratory work, there must always be considerable variation in the limits of our field classification, for there are many questions, such as climate, physiography, drainage, and farm practice, which are recognized as having large influences upon crop production which can not be recognized in a statement of a mechanical analysis. Considerable variation, therefore, may be allowed in the mechanical analysis of soils within the same type from which the same results may be attained, both as to character and yield of crops.

Very truly, yours,

Milton Whitney,
Chief Bureau of Soils.

The referee would merely add to this, that whoever attempts to determine the availability of plant food, or the crop-producing power of soils, be he chemist, soil physicist, or agronomist, needs all the data he can get; and if the chemist is going to attempt to say what a soil will do, or how it can be most economically improved, he should have methods for making the physical as well as the chemical examination.

Methods for the Analysis of Alkali Soils.

The points which have received attention are the effect of dilution on the amount of salt dissolved, and the titration methods for the determination of carbonates, bicarbonates, and chlorids. As our present methods for the determination of silica, sulphuric acid, and the bases are applicable here, no attention was given to these points. For this work 10 samples of alkali soils and crusts were selected, and prepared for analysis by passing through a 2-mm sieve. The samples represented soils containing large and small quantities of the acid radicals to be determined, as well as samples high and also lower in soluble lime compounds. The results obtained are given in the following table:

TABLE V.—*Effect of dilution on solubility of alkali soils in water.*

RESULTS BY J. F. BEAZEALE.

Dilution.	Constituents.	4721.	4724.	4686.	4759.	4889.	5055.	5102.	5103.	6315.	4782.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
50 grams per liter..	Ca	0.08	0.08	Trace.	1.65	0.02	0.03	0.25	Trace.	2.26	1.43
	Mg	.01	.01	Trace.	.08	Trace.	.01	.36	Trace.	.08	.67
	N	.46	1.58	9.39	5.12	22.70	.39	17.66	11.91	1.66	4.03
	K	.13	.14	.11	.28	.68	.05	.09	.07	.07	.06
100 grams per liter.	CO_3	....	.03	6.86	.02	13.40	.15	....	6.00	....	....
	HCO_3 ...	.23	.35	5.45	.05	13.93	.26	.08	3.57	.03	.08
	Cl	.47	1.41	1.76	6.29	.91	.35	18.01	3.60	5.54	5.96
	SO_4	.22	.76	1.32	3.60	.98	.04	5.14	5.21	.12	3.78
50 grams per liter..	CO_3	.03	.07	5.96	....	13.40	.17	....	5.84	....	....
	HCO_3 ...	.26	.45	6.85	.12	16.90	.14	.12	3.74	.07	.09
	Cl	.44	1.44	1.80	6.48	.84	.34	17.62	2.24	5.36	6.65
	SO_4	.19	.63	1.28	4.32	.99	.04	12.80	7.09	.09	5.49
25 grams per liter..	CO_3	.07	.07	5.76	....	12.47	.16	....	5.96	....	....
	HCO_3 ...	.52	.58	5.28	.17	16.43	.24	.19	3.86	.09	.14
	Cl	.50	1.40	1.96	5.34	.84	.34	16.14	2.56	5.22	6.20
	SO_4	.27	.77	1.67	6.34	1.12	.09	12.40	7.37	.13	7.06
10 grams per liter..	CO_3	.17	.23	6.48	....	13.00	.59	....	5.96	....	....
	HCO_3 ...	.72	.90	6.52	.24	15.22	1.20	.18	4.53	.24	.24
	Cl	.56	1.75	1.40	6.44	1.00	.35	16.28	2.52	5.47	6.61
	SO_4	.27	.70	1.20	9.95	1.04	.04	13.42	8.83	.12	9.11
RESULTS BY F. P. VEITCH.											
50 grams per liter..	CO_3	0.07	0.12	6.57	0.06	14.54	0.21	....	6.86	....	....
	HCO_3 ...	.31	.38	6.54	.07	15.26	.02	.12	5.16	.04	....
	Cl	.47	1.51	1.40	5.71	.96	.30	17.04	3.44	5.21	....
	SO_4	.17	.43	[a] .89 [a] 1.40	[a] 2.94 [a] 4.91	.73	.02	9.03 [a] 14.60	7.91 [a] 12.72	.18	....
10 grams per liter..	CO_3	.24	.24	6.91	....	14.66	.36	....	6.31	....	....
	HCO_3 ...	.72	.61	5.32	.30	15.22	.86	.30	5.92	.36	.24
	Cl	.56	1.40	1.40	5.88	.84	.42	17.20	3.50	5.32	6.56
	SO_4	.30	.86	1.39	10.52 [a] 12.04	1.04	.18	14.64 [a] 14.38	12.92	.12	10.18

[a] Determination made after ninety-six hours standing in contact with the soil.

The results may be summed up as follows: The concentration of the solution materially affects the rapidity of solution of sulphates, particularly calcium sulphate, and it is quite evident that equilibrium has not always been reached in twenty-four hours. The chlorin determination is the most satisfactory and appears to be the least affected by the concentration of the solution, while equilibrium is usually reached within the twenty-four hours. The determination of carbonates and of bicarbonates when present in large quantities has not been very satisfactory to the referee. This is attributed to the uncertain end point with phenolphthalein, which is far from sharp, particularly in the colored solutions produced by black alkali. The total titration of carbonates plus bicarbonates, using methyl orange, was somewhat more satisfactory.

While it may be true that the accurate determination of that portion of the soluble salts in excess of the quantity fatal to vegetation may not be of practical value so far as such soils are concerned at the present time, yet the possibility of reclaiming such lands and the importance of the composition of irrigating waters for this purpose make it highly desirable that we have a uniform and reliable method for this work. The referee, therefore, will recommend that this matter go to the referee next year, with the suggestion that the concentration of solution and time required for all salts to come to equilibrium receive further attention. It is to be hoped that those members whose location and work bring them in contact with this class of soils will give the matter some attention.

RECOMMENDATIONS.

It is recommended:

(1) That the N/200 HCl method be given further trial.

(2) That samples be so taken that it will be possible to determine by this method the available potash and phosphoric acid in each 12 or 9 inches to a total depth of 3 or 4 feet.

(3) That the methods for the analysis of alkali soils be referred to the referee with instructions to determine the most desirable amount of soil and the amount of water to be used in dissolving the soluble salts.

The **PRESIDENT**. If there is no objection the reading of the papers relating to soils will be deferred and we shall have the pleasure of listening to another guest of the association. Some thirteen years ago when I went to Europe it was my great pleasure to make the acquaintance of our highest authority on explosives, and it now gives me particular pleasure to present him in Washington, Professor Munroe, of Columbian University.

ADDRESS OF PROFESSOR MUNROE, OF COLUMBIAN UNIVERSITY.

GENTLEMEN. When I greeted your president and he extended to me an invitation to meet you this morning, my old friend, your honored secretary, informed me that it was the custom to make a speech when introduced, but I can not make a speech. While I feel complimented by being called upon, I am at a loss how to greet you. I would greet you in behalf of the university, but the president has done so in a most interesting manner. I would greet you in behalf of Washington, but there is no reason why I should do that, since Washington is the common home of us all. I have the privilege of being here to a greater extent than you, but Washington is the home of all Americans. I simply greet you as I would at your own home.

I would say that I have been interested in this organization since its beginning and have noticed the steps that were being taken and the great progress made. I feel very much pleased at what it has accomplished. I have been glad to call the attention of my students to the work of the association along the line of agriculture, and have urged them to attend the meetings of this body, to see you in person and to listen to these discussions. The accounts which I have read of these proceedings as I have received them from year to year have been very interesting, and I hope that I may continue to get them.

I have been much interested in your attempt to secure uniformity in methods. Many years ago at Newport, in purchasing materials for the Government, I was obliged to make specifications for them. There was difficulty in securing material to conform to the specifications at that time. Some twenty years ago I suggested that we append to the specifications the method by which the different constituents called for should be determined. It may be seen that the methods then proposed were not the most exact nor the best, but it was necessary that the analysts should follow the same method. The errors of the methods were eliminated and only the errors of the analysts remained. Your method of examining into the different methods proposed and then declaring that certain methods shall for the time be the official method seems to me to be most admirable. You are ready to change when the better method is proposed and this agreement is a marked progress. In order to obtain certain data it is necessary that you follow similar methods and, even if there is some variation in the data, it is not so unsatisfactory as it otherwise might be.

I repeat that I feel gratified at your having invited me to meet you, and I am glad to say that I shall be able to make another contribution other than the few words I have spoken. Someone has expressed a desire for the bulletin which has been published by the Census Bureau on Chemicals and Allied Products, and, so far as possible, I shall be glad to supply copies to your members. That will be my chemical contribution to this meeting.

The PRESIDENT. I am sure we appreciate the honor of having Professor Munroe with us, and in behalf of the association I extend to him our thanks for the donation of these publications.

REPORT OF COMMITTEE ON NOMINATIONS.

Mr. BIGELOW. We would nominate for vice-president Mr. M. E. Jaffa; for secretary, Mr. H. W. Wiley, and for the additional members of the executive committee, Messrs. Penny and Leitch. The recommendation includes the nomination for president of Mr. Davidson.

Mr. VAN SLYKE. I move that the report of the committee be adopted and that the secretary be instructed to cast the ballot for the officers designated.

The motion was carried and the officers were declared duly elected.

Mr. BIGELOW. I would like to say for the benefit of those who can go to Cabin John that we will be able to get a very nice dinner for $1, but it will not include wine or anything of that nature. I would like to know how many members can go. We will take the cars that pass in front of the Ebbitt House, and it is suggested that we start about 5 o'clock.

A QUANTITATIVE METHOD FOR DETERMINING THE ACIDITY OF SOILS.

By C. G. HOPKINS, W. H. KNOX, and J. H. PETTIT.

Although for many years we have been able to determine by qualitative methods whether a soil is acid, or sour, and although different degrees of acidity have sometimes been recognized and described by such terms as slightly acid, very acid, strongly acid, etc., no practical quantitative method for determining the exact degree of acidity and the consequent quantity of lime required per acre to correct or neutralize the acidity of the soil has been described so far as the authors have been able to learn. Certainly no such method is in common use.

The method which we have worked out is based upon the reaction which occurs when solutions of mineral salts are added to acid soils. The acids of the soils are themselves very difficultly soluble, and it is practically impossible to completely extract them from the soil with distilled water, even though large quantities of water be percolated through the soil; but, when a mineral salt solution is added to the soil, the acids apparently unite with the mineral base, evidently liberating the mineral acid, or an acid salt which, of course, is perfectly soluble, and whose titrating power furnishes a very satisfactory basis for determining the total acidity of the soil.

For use in quantitative work a 5 per cent solution of commercial sodium chlorid (a good grade of common table salt) has been found to be entirely satisfactory. Of course, other mineral salts, as potassium chlorid, serve the purpose equally well, but they are no better than common salt and are considerably more expensive.

The details of the method are as follows: Place 100 grams of the soil in a stout, medium wide-mouthed bottle of about 400-cc capacity; add a sufficient quantity of a 5 per cent sodium chlorid solution to make 250 cc of liquid, including the moisture contained in the soil, but independent of the volume of the soil itself (the moisture content of thoroughly air-dry soils will not cause an appreciable error, if neglected); close tightly with a rubber stopper; place in a shaking machine, and shake for three hours. (Shaking by hand every half hour for about twelve hours will accomplish the same result). Place the bottle in a suitable centrifuge and whirl till the soil is thrown down sufficiently to allow at least 125 cc of clear liquid to be blown off by means of two glass tubes held in a rubber stopper and arranged about the same as in the ordinary "spritz" bottle, except that the exit tube should be about 2 mm at the end instead of terminating in a fine jet. Instead of using the centrifuge the bottles may be allowed to stand a few hours, or 125 cc of the liquid may be filtered off, care being taken to avoid concentration by evaporation. Exactly 125 cc of the clear liquid (one-half the total volume of liquid) are placed in an Erlenmeyer flask, heated to boiling[a] to expel traces of carbon dioxid and to insure a sharp "end reaction," and then titrated with standard fixed alkali, phenolphthalein being used as the indicator.

The reaction between the soil acids and the sodium chlorid does not run to an end, but it has been found that at the end of three hours' shaking, under the conditions described above, an equilibrium is established, and that no appreciable additional quantity of acid is liberated with repeated or long-continued shaking. In other words, the solution has the same degree of acidity whether the titration is made as soon as possible after the three hours' shaking, or after shaking for three hours it is allowed to stand for twenty-four hours and then again shaken for three hours before drawing off one-half of the solution to titrate. If, however, after the 125 cc of liquid are removed and titrated, an equal quantity of fresh salt solution is added to the residue contained in the shaker bottle, the contents are again shaken for three hours, and 125 cc of the solution is drawn off and titrated, this second titer will be found to be approximately two-thirds of the first titration, instead of one-half of it, as it would be if the reaction had run to an end under the first treatment. If this process is further repeated, the third titration will be found to be about two-thirds of the second, and the fourth to be two-thirds of the third, etc. We are thus able by this fractional process to separate the acid equivalent from the soil to within an indeterminable amount, and the results obtained form a simple descending geometric series, as may be seen from the following data, in which are shown the duplicate readings (in cubic centimeters of standard alkali) of eleven different titrations of the acid obtained in these fractional separations.

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[a] No appreciable quantitity of hydrochloric acid is lost from such dilute solutions even when heated to boiling for a short time.

TABLE I.—*Standard alkali neutralized by successive saline extracts.*

Titration (soil sample No. 1.)	(A) Standard alkali.	(B) Standard alkali.	(Average.) Standard alkali.	Ratio to preceding titration.
	cc.	*cc.*	*cc.*	
First	8.9	9.1	9.0	....
Second	5.8	5.6	5.7	0.633
Third	3.9	3.5	3.7	.649
Fourth	2.7	2.6	2.65	.716
Fifth	1.9	1.9	1.9	.717
Sixth	1.25	1.25	1.25	.658
Seventh	1.0	1.0	1.0	.800
Eighth	.75	.75	.75	.750
Ninth	.5	.5	.5	.667
Tenth	.3	.3	.3	.600
Eleventh	.2	.2	.2	.667
Average ratio	....	....	....	.686

It will be observed that the ratio is fairly constant, considering the small amounts of liquid measured in the titrations.

The duplicate readings of the first and second titrations on four other samples of soil gave the following results:

TABLE II.—*Comparison of first and second soil extracts.*

Soil sample No.—	First titration.			Second titration.			Ratio of second to first.
	(A) Standard alkali.	(B) Standard alkali.	(Average.) Standard alkali.	(A) Standard alkali.	(B) Standard alkali.	(Average.) Standard alkali.	
	cc.	*cc.*	*cc.*	*cc.*	*cc.*	*cc.*	
2	18.1	18.6	18.35	12.6	12.2	12.4	0.676
3	5.7	5.6	5.65	3.7	3.75	3.73	.660
4	4.8	5.2	5.00	3.0	3.2	3.1	.620
5	2.8	2.8	2.8	1.8	1.9	1.85	.661
Average ratio	....	....	....	....	....	....	.654

If we take an average of these two general averages, we have 0.67, or approximately the fraction ⅔. We are investigating still further this ratio, or the relation of each titration reading to the preceding one, but tentatively we are using this ratio and believe it is very approximately correct. By computation from these data, it becomes a simple matter to obtain a factor by means of which we can compute the total acidity from the first titration. For example, if we let X equal the constant ratio, S the total acidity, and 1 the first titration, we have the following equation:

$$S=1+X+X^2+X^3+X^4+\ .\ .\ .\ .\ .\ X^n$$

$$\text{or } S=\frac{1}{1-X}$$

$$\text{If } X=\tfrac{2}{3}\text{, then } S=\frac{1}{1-\frac{2}{3}}=3.$$

Thus, if ⅔ is the correct ratio of each titration to the preceding one, then the total acidity of the soil is three times the first titration. From the data which we have thus far obtained, we believe this factor to be very nearly exact; and in practical work we recommend that only the first titration be made as above described and that this reading be multiplied by the factor 3 to determine the total acidity in the sample of soil taken.[a] If we apply this rule to soil No. 1, we have 26.7 cc and 27.3 cc, or an average of 27 cc, based upon the first titration only; whereas if we add together all titrations and then add to that sum 0.4 cc, or twice the amount of the eleventh titration, we have 27.6 cc and 27.1 cc, or an average of 27.35 cc, based on fractional separations and absolute titration of the total acidity excepting the 0.4 cc in the final residue.

It is convenient to have the strength of the alkali such that 1 cc is equivalent to 3⅓ mgs of calcium carbonate, so that the number of cubic centimeters of alkali used in the titration corresponds to the number of centigrams of calcium carbonate required to neutralize the acid in 100 grams of the soil sampled. In other words, each cubic centimeter of alkali required for the single titration corresponds to one-hundredth of 1 per cent of the soil in terms of calcium carbonate required By computation from this datum and from the apparent specific gravity of the soil, as used in the determination, and the thickness of the stratum sampled, the exact amount of calcium carbonate required to correct the acidity of the soil is readily determined; and 56 per cent of this amount gives the quantity of fresh burned lime required, in case it is desired to use this form of lime.

If we can make our titration exact to one-tenth of a cubic centimeter of the standard alkali, which corresponds to one-thousandth of 1 per cent of calcium carbonate required by the soil to neutralize its acidity, the limit of error, or perhaps we might better say, the limit of accuracy, is 3 pounds of calcium carbonate per acre-inch of soil or 36 pounds per acre-foot, on the basis of 3,600,000 pounds of soil to the acre-foot; and we can compute the amount required by 40 acre-inches, or by an acre-meter, if we may use the term, to within a limit of error of 120 pounds of calcium carbonate or 70 pounds of lime.

To indicate the ease and rapidity of manipulation of this method, it may be said that with the shaking machine which we are using and without the use of a centrifuge, one analyst can easily determine quantitatively, and in duplicate, the degree of acidity in 30 different samples of soil in one day.

That the quantitative determination of the lime required by soils is of great practical significance, is evidenced by the fact that we find exceedingly marked differences in the needs of different soils, as may be seen from the following tabular statement:

TABLE III.—*Data employed in the determination of the acidity or lime requirement of soils.*

Field or plot tested	Name of stratum sampled.	Location of stratum.	Apparent specific gravity of soil.	Weight of stratum per acre.	Alkali required for the titration.		$CaCO_3$ required by soil.	$CaCO_3$ required for stratum per acre.
		Inches.		*Pounds.*	*cc.*	*cc.*	*Per cent.*	*Pounds.*
A........	Surface............	0-6	1.326	1,798,000	1.9	1.7	0.018	324
	Subsurface	6-19	1.349	3,963,000	7.2	7.2	.072	2,853
	Subsoil	19-40	1.456	6,910,000	14.4	14.7	.146	10,088
B........	Surface............	0-7	.726	1,149,000	1.7	1.7	.017	195
	Subsurface	7-17.5	1.278	3,032,000	....	....	....	....
	Subsoil	17.5-40	1.299	6,605,000	....	....	....	....
C........	Surface............	0-5	1.227	1,387,000	8.7	8.5	.086	1,193
	Subsurface	5-20.5	1.256	4,400,000	11.7	11.9	.118	5,192
	Subsoil	20.5-40	1.354	5,967,000	31.1	31.3	.312	18,617

[a] Since this paper was read some additional data have been obtained which show that the factor 3 is too small. Further results will be offered for publication within a short time. In practical work we now advise using the factor 4, which will probably prove to be above the exact figure, but in practice it is advisable to apply at least a small excess of lime above the actual present requirements of the soil.—C. G. HOPKINS.

TABLE III.—*Data employed in the determination of the acidity or lime requirement of soils*—Continued.

Field or plot tested.	Name of stratum sampled.	Location of stratum.	Apparent specific gravity of soil.	Weight of stratum per acre.	Alkali required for the titration.		CaCO$_3$ required by soil.	CaCO$_3$ required for stratum per acre.
		Inches.		*Pounds.*	*cc.*	*cc.*	*Per cent.*	*Pounds.*
D........	Surface............	0–6	1.291	1,751,000	0.6	0.6	0.006	105
	Subsurface	6–24	1.315	5,349,000	.3	.4	.004	214
	Subsoil	24–40	1.328	4,802,000	1.9	1.9	.019	912
E........	Surface............	0–7	1.307	2,068,000	.6	.6	.006	124
	Subsurface	7–17	1.368	3,092,000	.3	.3	.003	93
	Subsoil	17–40	1.408	7,319,000	....	....	....	....
F........	Surface............	0–7	1.238	1,958,000	6.6	6.8	.067	1,312
	Subsurface	7–16	1.256	2,554,000	14.9	14.9	.149	3,806
	Subsoil	16–42	1.364	8,012,000	9.3	9.5	.094	7,531
	Lower subsoil	42–54	1.357	3,679,000	.1	.1	.001	87

It will be observed that some soils are exceedingly acid, and that they will require exceedingly heavy applications of some form of lime to produce a neutral condition. The soil in field A requires 324 pounds of calcium carbonate per acre to neutralize the surface, or plowed soil; 2,853 pounds to neutralize the subsurface, or that which lies between the plow line and the subsoil line, and 10,088 pounds to correct the acidity of the subsoil to a depth of 40 inches. The total amount required by this soil to neutralize the acidity to a depth of 40 inches is 13,265 pounds of calcium carbonate, or about 6½ tons. This is equivalent to about 3½ tons of burned lime.

It should be stated that we do not know as yet to what depth it is necessary to neutralize the soil acidity. We hope to have some definite information on this point within a few years. It is certainly safe to say that the acidity of the plowed soil should be corrected, and it seems very probable that it will be advantageous to apply some excess above what is required for that purpose. We are carrying on some experiments to determine how rapidly lime is carried into the lower depths of the soil; or, in other words, how much time will be required before lime which is mixed with the surface soil will begin to reduce the acidity of the subsurface or subsoil.

The soil of field B is very slightly acid, the total amount of calcium carbonate required being only 195 pounds per acre, and this is all needed in the surface soil. Indeed, both the subsurface and subsoil of this field were found to contain lime carbonate, and all that is needed to correct the slight acidity of the surface soil is somewhat deeper plowing than has been practiced heretofore.

Field C is possessed of one of the most acid soils which we have found in the course of several hundred examinations of acid soils. The plowed soil requires 1,193 pounds and the subsurface 5,192, while to correct the total acidity of this soil to a depth of 40 inches requires 25,000 pounds, or 12½ tons of calcium carbonate, equivalent to 7 tons of burned lime.

Field D possesses a rather small degree of acidity, 1,231 pounds of calcium carbonate, or 689 pounds of burned lime, being sufficient to neutralize the soil acidity to a depth of 40 inches.

Field E requires a total of only 217 pounds of calcium carbonate. The subsoil was found not only free from acidity, but it contains abundance of calcium carbonate to correct the acidity of the upper strata; but, as there is no practical method of effecting such a translocation, it is necessary to apply some form of lime to the surface.

It will be noted that there are not only marked differences in the degree of acidity of acid soils, but the acidity of distinctly acid soils is markedly different in different strata of the same soil; and, furthermore, the acidity increases with depth in some soils and decreases in others, while in field F the acidity increases from the surface to the subsurface, and then grows less in the ordinary subsoil, practically disappearing in the lower subsoil below 42 inches. This condition is probably explained by the fact that the subsoil stratum lying between 16 and 42 inches in depth is of a stiff clayey nature and nearly impervious to water, and the soil acids of the upper strata have not been carried through it to any appreciable extent. This field requires a total of 12,686 pounds of calcium carbonate, or 7,104 pounds of burned lime, to correct the acidity of the soil, and it may be noted that this applies to the total depth of the soil in this field.

It may be pointed out that there are several advantages of considerable economic value in being able to apply lime quantitatively in correcting the acidity of soils; among these advantages may be mentioned the following:

1. One may be sure that he is applying sufficient lime to correct the acidity and thus really benefit the soil.
2. One may avoid a large waste of lime by applying an ample amount without a large excess.
3. One may correct the acidity of the soil without danger of the possible injury to the land which is thought to be sometimes produced by excessive applications of caustic lime.

In our own work we already have illustrations of some of these advantages. For example, an application of only 320 pounds per acre of air-slacked lime to a very slightly acid soil effected an increase of more than one-half ton of air-dry alfalfa hay per acre for each of the first two cuttings this season, while somewhat heavier but nevertheless insufficient applications on exceedingly acid soils have produced no apparent benefit, and, in fact, have led people who are not cognizant of the strongly acid character of the soil, to suppose that the soil does not need lime.

In conclusion, it is desired to state that this method of making quantitative determinations of the acidity or lime requirement of soils has now been in almost constant use for nearly a year in the laboratories of the University of Illinois Agricultural Experiment Station. Quantitative applications of lime have been made to soils in the laboratory in accordance with the determinations made by this method, and the acidity of the soil has been found to have been practically neutralized. The method has been applied to pot-culture investigations and also to field experiments on a large scale, involving carloads of the material, and it is asserted with confidence that it can be relied upon to give trustworthy results. A brief description of this method was given before the Illinois Farmers' Institute in February of the present year. It has also been described to a number of other people interested in the study of soils, including several who are engaged in soil investigations in the United States Department of Agriculture, and it is gratifying to state that it has met with very general approval. The authors beg to report the method to this association with the hope that it may add to the satisfaction and encouragement which we all find in anybody's discovery or development of any method or principle which materially assists in reducing agricultural practice to a scientific basis.

The PRESIDENT. The eyes of this association as well as all those interested in agriculture are always upon the Secretary of Agriculture. From my own knowledge of the work that has been done for the agricultural interests of this country, I would say that no Secretary has ever grasped the situation so completely or has been able to accomplish so much for the agriculture of this country as our present Secretary. It gives me, therefore, the utmost pleasure to introduce to you the honorable Secretary of Agriculture, Mr. James Wilson.

ADDRESS OF SECRETARY WILSON.

Mr. President and Gentlemen of the Association: I came here merely to take off my hat and welcome you to the city, and to congratulate you on the good work you are doing in the interest of our country. There are few people who know how fast the United States is progressing along all scientific lines. I hardly know of anyone who makes a special study of the matter; it keeps me busy to keep up with my own Department. The kind words just spoken by your president concerning myself require a word of explanation. I try to get the right man for the right place, and after I have found that man I take the check line out of the hook and give him a free head and he brings things to pass.

You never had a Secretary who understood all the sciences. One might spend fifty years studying them and then find that he was fifty years behind and that he must begin all over again, and so on until he was too old to learn. I have gotten over the notion that I must understand the details of every scientist's work. I try to study the scientist. I think the Department of Agriculture has as fine a corps of scientists as can be found on the face of the earth. As we progress, grow, and develop in the United States, we need chemists. We are needing them more and more now to safeguard the health of our people, and it is necessary to have their assistance that the people shall not be poisoned and that they shall not be defrauded by the quality of the food they necessarily consume.

The manufacturers' work grows and develops just as fast as the chemists help them along and no faster. For drugs, for example, we are heavily indebted to Europe and pay heavily every year. Scientists of the Department of Agriculture are about to undertake bringing into our country all the drug plants used in the work in chemistry. That will help along this one line. I have one determination which I hold strongly, and that is that whenever I see Uncle Sam paying for anything from foreign countries I want to find out whether that thing can not be produced at home. Four years ago we manufactured from beets 16,000 tons of sugar, the next year 41,000 tons, the year after that 72,000 tons, and the year after that 184,000 tons. This year it will be over 250,000 tons. We do not want the United States to pay out money for things which our people can produce at home. I may safely say before this association of chemists that sugar is composed of wind and water. Four years ago we produced a fourth of the rice that was consumed in the United States. This year all the rice needed in America, except some special brands, will be produced in this country, and so it goes all along the line. We are endeavoring to grow the things for which we are paying heavily to foreign countries. I do not think it is necessary for us to leave their production to other lands. We have every degree of climate and widely varying conditions from Alaska on the north to the Philippines on the south, and we can produce everything we want. It is the purpose of the Secretary of Agriculture that we shall progress along these lines until whatever we need can be produced somewhere in the United States. In all of these investigations we shall need chemists, and we are going to need them very much.

We have been selling $30,000,000 worth of home-grown tobacco and buying $6,000,000 worth of fine Sumatra tobacco. We have investigated and experimented until now we are able to help the people of New England to produce this fine tobacco. It will only be a question of time when we shall keep that 6 millions of dollars at home. We have determined to produce filler tobacco. We have been trying to find out the climate best adapted to its culture and now we have found two places in the United States where we can grow the finest tobacco under the sun. We find other places where it is only a little too strong; there we shall institute scientific research and grow the tobacco under all possible variations of conditions in order to get just what we want, and while we are doing this we want chemists to watch the plant's progress from the time it takes root. It is wonderful what the American chemist can find out. This work with tobacco means the annual saving of about 14 millions of dollars that we are now paying out for foreign-grown tobacco. We can eventually produce all this much better at home and supply the world. The prosperity of the United States will be the result of the labors of the American people.

I am obliged to you for sending the committee to wait on me. I hope your stay here will be pleasant and that you will arrive home safely. I bid you good day.

The **President**. Is it your pleasure that we continue with our program, or shall we adjourn?

Mr. **Penny**. I would like to ask the referee on soils if he found any of the surface soils that were not acid?

Mr. **Hopkins**. No, sir.

Mr. **Penny**. How did you determine that they were acid?

Mr. **Hopkins**. By the method I have just described for determining the acidity of soils, and also by making a determination of the carbonates or carbon dioxid.

Mr. **Penny**. Did you make the acidity test upon the whole soil, or only on part of it?

Mr. **Hopkins**. Ordinarily, we separate material coarser than 1 mm and make our determination upon the fine earth.

Mr. **Fraps**. If there is no objection, I would like to present this paper; it will take only about ten minutes.

STUDIES IN NITRIFICATION.

By G. S. Fraps.

At the last two meetings of this association articles on nitrification have been presented by W. A. Withers and the author. The chief fact brought out in these articles was that in three weeks some soils nitrify ammonium sulphate to a greater extent than cotton-seed meal does, while with other soils the reverse is the case. The explanation for this circumstance advanced in the articles referred to is that the difference is due to the combined influence of three factors:

1. Ammonium sulphate may hinder the action of the nitrifying organisms.
2. The acids produced by their life activity are detrimental to them unless neutralized.
3. There are organisms which convert organic matter directly to nitrites, as well as those which oxidize ammonium salts to nitrites.

It was to test this last hypothesis that the work I am about to describe was instituted. The results obtained at first were so astonishingly irregular that the success of the investigation seemed to be threatened. A search for the cause of the irregularities led, however, not only to some very interesting results in regard to the life history of the organisms, but paved the way for a study of the nature of the nitrifying organisms and laid the foundations for a rational method for studying the nitrifying powers of soils.

Method of Work.

Two kinds of nitrification tests were made. In one kind the nitrification was carried out in the soil as it came from the field; in the other the tests were carried out in a sterile soil inoculated with an active soil. The first will be called direct nitrification tests; the second, inoculation tests. A large number of both kinds of tests were made in the course of this investigation. The methods used varied slightly at first, but finally the following were adopted as the standard methods:

1. DIRECT NITRIFICATION TESTS.

The sample of soil, as received, was sifted through a 5-mm sieve and mixed thoroughly. Water was determined in it by the usual method, and also its water saturation capacity, as follows:

Fifty grams of the soil were placed in a carbon filter on a perforated porcelain plate, and the whole weighed. Water was added in quantity a little more than was necessary to saturate the soil, so that a few drops came through, and the tube was allowed to drain, a cork being placed in the mouth of the tube to prevent evaporation from the surface of the soil. After two hours the stem of the tube was dried, and the whole weighed. Allowance was made for the water already in the soil.

In all the nitrification tests 500 grams of soil were used and the quantity of cotton-seed meal, or ammonium sulphate, which contained 0.3 gram nitrogen, together with 2.14 grams calcium carbonate, which is a sufficient quantity to neutralize all the nitric acid and sulphuric acid which would be formed if the ammonium sulphate were completely nitrified. The soil was placed in a porcelain evaporating dish and mixed intimately with the cotton-seed meal (4.30 grams) and calcium carbonate. Sufficient water was then added to bring the water content of the soil to one-third of its saturation capacity, and the whole thoroughly mixed. The mixture was transferred to a pint precipitation jar, or fruit jar, weighed, placed in a water bath in a dark closet, and kept as nearly as possible at 35° C. Every Monday and Thursday the jars were weighed again, and the loss of water replaced in each one. At the end of the period of three weeks the contents of each jar were mixed thoroughly and 100 grams weighed out, extracted with water, and the nitrates determined by the Tiemann-Schulze method. The remainder of the soil was returned to the jar and allowed to nitrify. Nitrates were determined at other periods, as a rule.

In case ammonium sulphate was used for the nitrification the calcium carbonate was mixed with the soil, and water added if necessary. Twenty cubic centimeters of a solution of ammonium sulphate, containing 1.42 grams of the salt, were then added, and the soil was thoroughly mixed. In every case the total amount of water present was kept at one-third of the water saturation capacity of the soil.

It must be noted that the 100 grams of soil taken for analysis did not represent one-fifth of the soil, since the total weight of the soil was 500 grams plus the weight of calcium carbonate, water, and nitrogenous substances. Correction has been made for this fact wherever necessary, but in comparing nitrification in different samples of the same soil no correction has been made, since the same quantity of material was present in each case.

INOCULATION EXPERIMENTS.

For the inoculation experiments a sandy clay soil was used, which had been in the laboratory for three years in the dry condition. It was further sterilized by treatment with chloroform, followed by a drying in an air bath. Its water saturation capacity was 34.3 per cent; so 11.4 per cent water was maintained in it during the tests.

Twenty grams of the soil used for inoculation was mixed with calcium carbonate, and with cotton-seed meal, if it was to be used, in a porcelain dish. The mixture was added to 500 grams of the sterilized soil, mixed thoroughly, a sufficient quantity of water added to bring the water content of the soil to one-third of its water saturation capacity, and the mixture stirred until it appeared uniform. It was then placed in jars, and the experiment conducted as described above.

When ammonium sulphate was used, the soil used for inoculation was mixed with calcium carbonate, and then with the soil. Water was then added, and finally 20 cc of the solution of ammonium sulphate already referred to. It was considered possible that the strong solution of ammonium sulphate might destroy some of the nitrifying germs if it came in contact with them before it was diluted.

In most of the experiments described in this article, the tests were carried out in duplicate. In many cases the agreement between the duplicates was satisfactory, but at times a jar would deviate from the normal. In such cases due judgment must be used in selecting the results. It can not be expected, in dealing with microscopic organisms, which are liable to vary in their activity, and sometimes for no known reason, that the same agreement can be obtained as in ordinary chemical analysis.

DESCRIPTION OF SOILS.

1746. Sandy clay soil from a wheat field on college farm; little humus; saturation capacity, 33 per cent.

1746a. A second sample, taken two months later.

1747. Sandy clay soil, sterilized as described previously; used in all inoculation experiments.

1748. Dark loam soil from a peach orchard on the experiment station farm; contains humus; saturation capacity 34 per cent.

1764. Black sandy soil from the Florida experiment station; contained only 0.5 per cent of water when it arrived, and had little nitrifying power.

1764a. A second sample, which had been kept in a well-closed fruit jar since the summer of 1901, and was still quite moist.

1766. Loam from a flower bed on college lawn; contains much humus.

1766a. A second sample, taken three weeks later.

1768. A garden soil.

1772. Sandy soil from the test farm of the North Carolina department of agriculture at Tarboro, N. C.; contained only 1.85 per cent water when received, but appeared moist; saturation capacity, 27.3 per cent.

1779. Poor, light, sandy soil from college farm; no humus; saturation capacity, 27 per cent.

1780. Rich, dark, sandy soil from college farm; contains much humus; saturation capacity, 46 per cent.

1781. Black, sandy soil from garden of State Chemist B. W. Kilgore, at Raleigh, N. C.; contains much humus; saturation capacity 42.5 per cent.

Acknowledgment is hereby made to Chemist H. K. Miller, of Florida, and State Chemist B. W. Kilgore, of North Carolina, for their kindness in furnishing samples of soil.

VARIATIONS IN THE NITRIFYING POWERS OF SOILS.

The irregularities referred to in an earlier part of this paper consisted in variations in the nitrifying power of the same soil. Samples taken at one time would nitrify vigorously, while another sample, taken a little later, would hardly nitrify at all, under the same conditions. For example, a soil taken from a wheat field May 28 nitrified 143 mg of nitrogen per kilo of soil in three weeks, while a sample of the same soil taken from the same place three weeks later nitrified only 20 mg in the same time.

Similar irregularities were observed in inoculation tests with soils. These variations are due to variations in the number and in the activity of the germs contained in the soil. The causes of the variations appear to be as follows:

1. *Variations in the water content of the soil.*—It is a well known fact that a decrease of the moisture of a soil below a certain limit is prejudicial to the activity of the nitrifying organisms. If the soil dries out, the organisms die, or become inactive; if it is moist, they thrive and increase.

2. *Variations in temperature.*—Prolonged cold decreases the numbers of the nitrifying organisms, while warmth is beneficial, and causes them to multiply.

3. *Abundance of food.*—The number of nitrifying organisms in different soils would vary somewhat with the food supply for them in the soil.

4. *Periodicity of the organisms.*—When the progress of nitrification is studied in soils under uniform conditions of moisture and temperature and plant food, it is seen that there are periods of activity of nitrification, preceded and followed by periods of comparative rest, during which much smaller quantities of nitrates are produced. This periodicity will now be discussed.

PERIODICITY OF NITRIFICATION.

In a number of experiments conducted during the course of this investigation, the rate of nitrification was studied by making determinations of nitrates in the soils at the end of three weeks, and at the ends of succeeding weeks. The quantity of nitrogen oxidized per week during each period shows the rate of nitrification in the soil. It is easily seen from a study of the results that the activity of the organism is periodical, and that a period of activity is preceded and followed by a period of comparative inactivity. The periodicity of the rate of nitrification of ammonium sulphate and cotton-seed meal is shown very strikingly in the four curves in figure 3. The periods are plotted as abscissae, and the number of milligrams of nitrogen oxidized

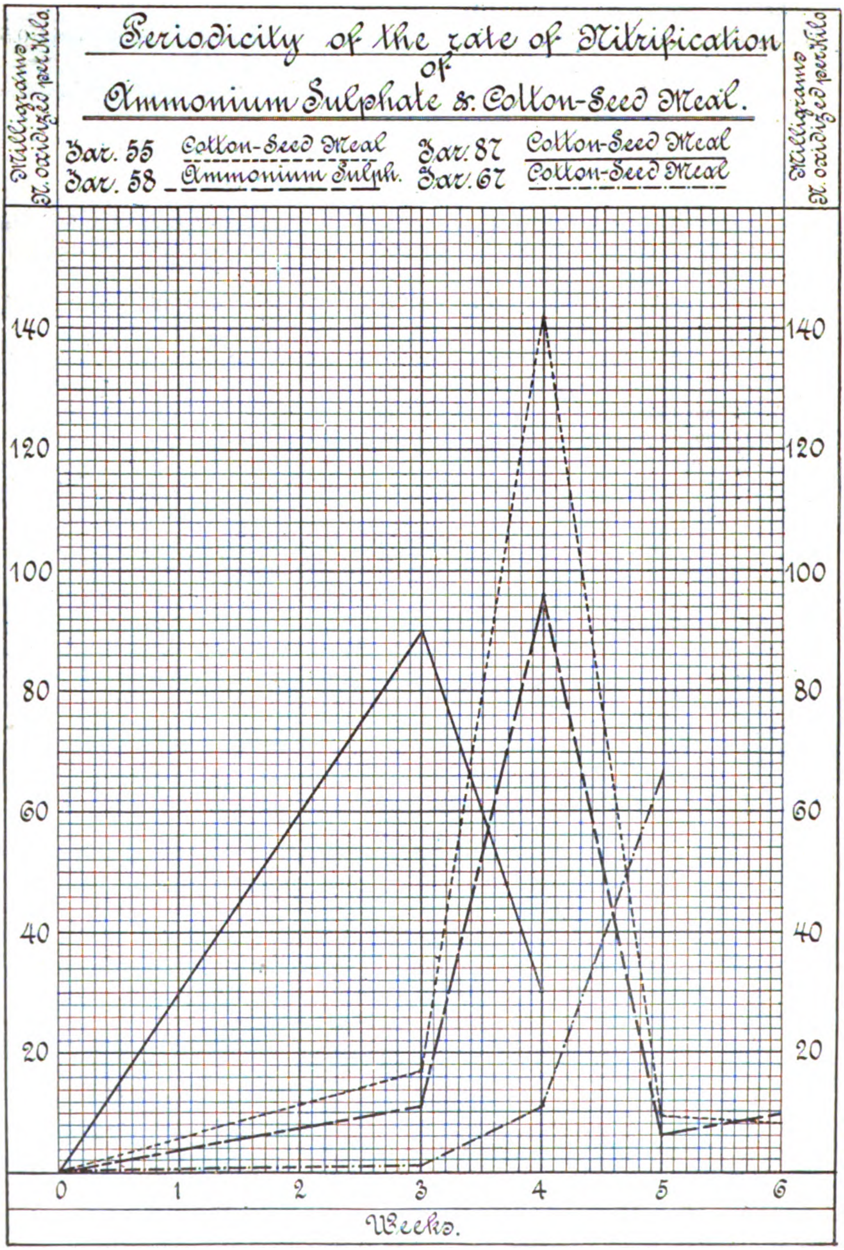

FIG. 3.—Periodicity of the rate of nitrification of ammonium sulphate and cotton-seed meal.

per kilo of soil per week during each period as ordinates. The curves were chosen to show how the period of greatest activity of nitrification may come during the first three weeks, or during the fourth week, or the fifth week, according to the condition of the soil at the time of starting the experiment.

Table I exhibits the rate of nitrification in 21 jars of soil, conducted in nine sets, and with organisms derived from seven different soils.

TABLE I.—*Rate of nitrification.*

[Nitrogen per kilo of soil oxidized per week.]

Soil and nutrient.	First three weeks.	Fourth week.	Fifth week.	Sixth week.	Total.
	Mgs.	*Mgs.*	*Mgs.*	*Mgs.*	*Mgs.*
Soil 1748, inoculation tests:					
Jar 55, cotton-seed meal	17	141	9	8	210
Jar 56, ammonium sulphate	5	59	....	[a] 37	138
Jar 57, cotton-seed meal	12	109	[b] −57	5	91
Jar 58, ammonium sulphate	11	96	6	9	143
Soil 1764, inoculation tests:					
Jar 67, cotton-seed meal	2	94	34	....	133
Jar 68, ammonium sulphate	2	53	8	....	69
Soil 1766, inoculation tests:					
Jar 72, cotton-seed meal	10	106	0	....	135
Jar 74, ammonium sulphate	2	61	[b] −9	....	57
Soil 1772, direct nitrification:					
Jar 77, cotton-seed meal	2	63	....	....	70
Jar 79, ammonium sulphate	1	1	0	....	4
Soil 1772, inoculation tests:					
Jar 81, cotton-seed meal	7	108	....	....	130
Jar 83, ammonium sulphate	3	62	....	....	70
Soil 1781, inoculation tests:					
Jar 91, cotton-seed meal	31	66	....	....	159
Jar 93, ammonium sulphate	25	49	....	....	124
Soil 1781, direct nitrification:					
Jar 85	7	5	....	....	26
Jar 87, cotton-seed meal	90	30	....	....	300
Jar 90, ammonium sulphate	93	39	....	....	319
Soil 1746, direct nitrification:					
Jar 47, cotton-seed meal	12	....	[c] 69	42	218
Jar 49, ammonium sulphate	7	....	[c] 28	52	129
Soil 1768, inoculation tests:					
Jar 62, cotton-seed meal	1	13	67	....	83
Jar 65, ammonium sulphate	2	9	64	....	79

[a] Mean of fifth and sixth week. [b] Denitrification took place. [c] Mean of fourth and fifth weeks.

An examination of the table brings out the following facts:

1. The rate of nitrification reaches a maximum, and then decreases.
2. The decrease in the rate of nitrification is not due to lack of nitrogen to nitrify. The soil contains 532 mg of nitrifiable nitrogen per kilo. The total amount of nitrogen oxidized in each jar is given in the table, and is less than half of the amount present in the soil in most cases.
3. The period of greatest energy of nitrification has come, as a rule, in the fourth week. This is the case with six of the nine sets of soils given in Table I. In one set it came in the fifth week, in one set during the first three weeks, and in one during either the fourth or fifth week.

4. The period of greatest energy of nitrification comes at the same time, under the same conditions, whether the nitrogen to be oxidized is supplied by ammonium sulphate or by cotton-seed meal.

5. The quantity of nitrogen oxidized during the first three weeks of a nitrification experiment depends on the activity of the organisms in the soil taken for the experiment, as well as on the number of germs present. In the same soil, and under the same conditions, it may happen that a sample taken at one time will oxidize a large quantity of nitrogen during the first three weeks, while a later sample will not nitrify to any great extent during that time, but will do so if kept a little longer.

CAUSE OF PERIODICITY.

The explanation of the variable rate with which nitrification takes place from time to time is to be found in the life history of the nitrifying organisms. There seems to be a period of multiplication of the germs, during which little nitrification takes place, then a period of multiplication and nitrification, followed by a period of multiplication without nitrification again. The reasons for this last statement will be developed later. After a time the number of nitrifying organisms no longer increases, but diminishes, on account of the consumption of nitrifiable matters. How long a period of time it takes for a group of the nitrifying organisms to complete this cycle of changes can not be stated. The maximum period of time required in any of the experiments here given for the maximum of activity of nitrification to occur was five weeks.

The life history of the nitrifying bacterium has a parallel in that of the bacteria which assimilate nitrogen on the roots of legumes. Cultivated outside of the plant, it has not yet been found possible to cause them to assimilate more than 7 mg per gram of sugar consumed by them, a very small quantity. When growing in nodules on leguminosæ, according to Nobbe and Hiltner, the bacteria are in the nature of parasites until they are transformed into bacteriod forms, and then the plant begins to receive the benefit of nitrogen assimilated from them. There thus appears to be two stages in the life of this organism, first a period of multiplication with little assimilation of free nitrogen, then a period in which the form of the bacteria changes, and assimilation of nitrogen takes place. As was stated, this life history is parallel to that of the nitrifying organisms.

INOCULATION ENERGY OF SOILS.

By the inoculation energy of a soil, we mean its power to induce nitrification in a sterile soil provided with nitrogenous substances and kept under favorable conditions. For the measure of the inoculation energy of a soil there has been taken the quantity of nitrates formed in three weeks in the sterile soil used throughout these experiments, inoculated with the soil to be tested. The method of conducting inoculation tests has already been described. The inoculation energy of a soil depends upon the number of germs in the soil and on their activity. Any influence which decreases the number of germs in a soil diminishes its inoculation energy.

The inoculation energy of certain soils or cultures was studied in two ways. The first experiments consisted in a comparison of the inoculation energy of the soil with the quantity of nitrates formed in it during the three weeks previous to starting the inoculation test, and were as follows: The soils in Series I were allowed to nitrify three weeks and the quantity of nitrates in them determined. Series II was then prepared by inoculation from Series I. At the end of a second three weeks Series II was analyzed and inoculations prepared from it, forming Series III. It should be stated that the soils in Series I were all inoculation tests, so that the soils used throughout the experiments are exactly the same, and the results are directly comparable. The results are given in Table II.

TABLE II.—*Inoculation energy of soils.*

[Nitrogen oxidized per kilo of soil.]

Nutrient.	Series I.	Series II.[a]	Series III.[a]	Ratio I: II.	Ratio II: III.
	Mgs.	*Mgs.*	*Mgs.*	*Mgs.*	*Mgs.*
Cotton-seed meal	182	52	81	3.5:1	0.6:1
Ammonium sulphate	176	15	4	12:1	3.8:1
Cotton-seed meal	73	35	12	2.1:1	2.9:1
Ammonium sulphate	67	32	25	2.1:1	1.3:1

[a] Series II represents the inoculation energy of Series I at the end of three weeks; Series III the inoculation energy of Series II.

The ratio of Series I to Series II, and of Series II to Series III, shows the effect of dilution of the actively nitrifying soil with 25 times its weight of sterile soil, and providing a quantity of nitrogenous matter. The series was not carried out in duplicate, and some of the variations are probably due to a jar going wrong. Excluding the greatest variations, the ratio of the quantity of nitrogen oxidized in the first three weeks to the quantity oxidized in the soil inoculated from it in the succeeding three weeks varies from about 2:1 to 3:1. As will soon be seen, however, this ratio depends on the activity of the inoculating soil or on the number of organisms present in it.

The second method of studying the inoculation energy of soils consisted in determining the inoculation energy of several soils at different periods during nitrification in order to see what change it undergoes. Four jars of soil were studied for the main experiment. They were kept under standard conditions and nitrates determined in samples taken at the end of three, four, five, and six weeks, respectively. At the end of three weeks Series II was prepared by inoculation from Series I. At the end of four weeks Series III was prepared from Series I, and Series IV was prepared at the end of the fifth week. The quantity of nitrogen in the form of nitrates in the soils of Series II, III, and IV at the end of three weeks represents, therefore, the inoculation energy of the soils of Series I at the end of different periods of time. The unbroken curves in figures 4 and 5 show the change in the inoculation energy of the four samples of soils with time. The inoculation energy of the soils increases regularly with the time during which nitrification takes place. It might be imagined that the inoculation intensity of a soil would vary somewhat as its energy of nitrification, but this is not the case, as may be seen by comparing figures 3 and 4. During the fifth week the nitrification intensity of the soil is very much lower than during the fourth week, while its inoculation intensity is higher than before.

There seems to be a very close relation between the inoculation intensity of a soil and the total quantity of nitrogen oxidized in it, as can be seen by comparing the unbroken curves in fig. 4 with the broken curves, which represent the total quantity of nitrogen oxidized. Even in the case in which denitrification took place (jar 57) the inoculation intensity of the soil exhibits a decrease corresponding with the decrease in the total quantity of nitrates in the soil. Table III contains the figures which were obtained in the inoculation experiment, and also gives the results of an experiment on soil No. 1781, which leads to the same conclusion as the other one. It gives the inoculation energy of the soils at the end of each period, and also the total quantity of nitrates present in them.

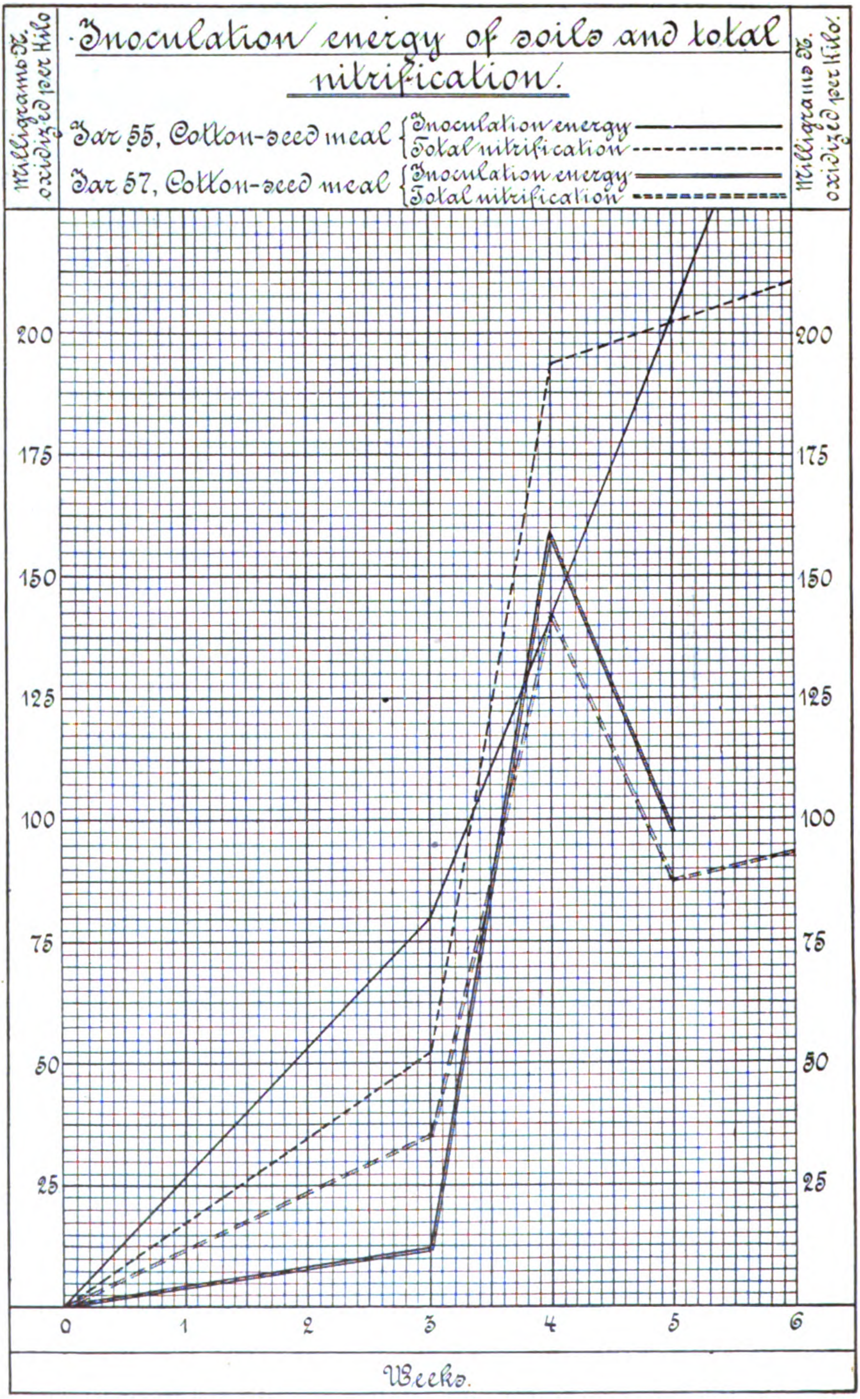

FIG. 4.—Inoculation energy of soils and total nitrification (cotton-seed meal).

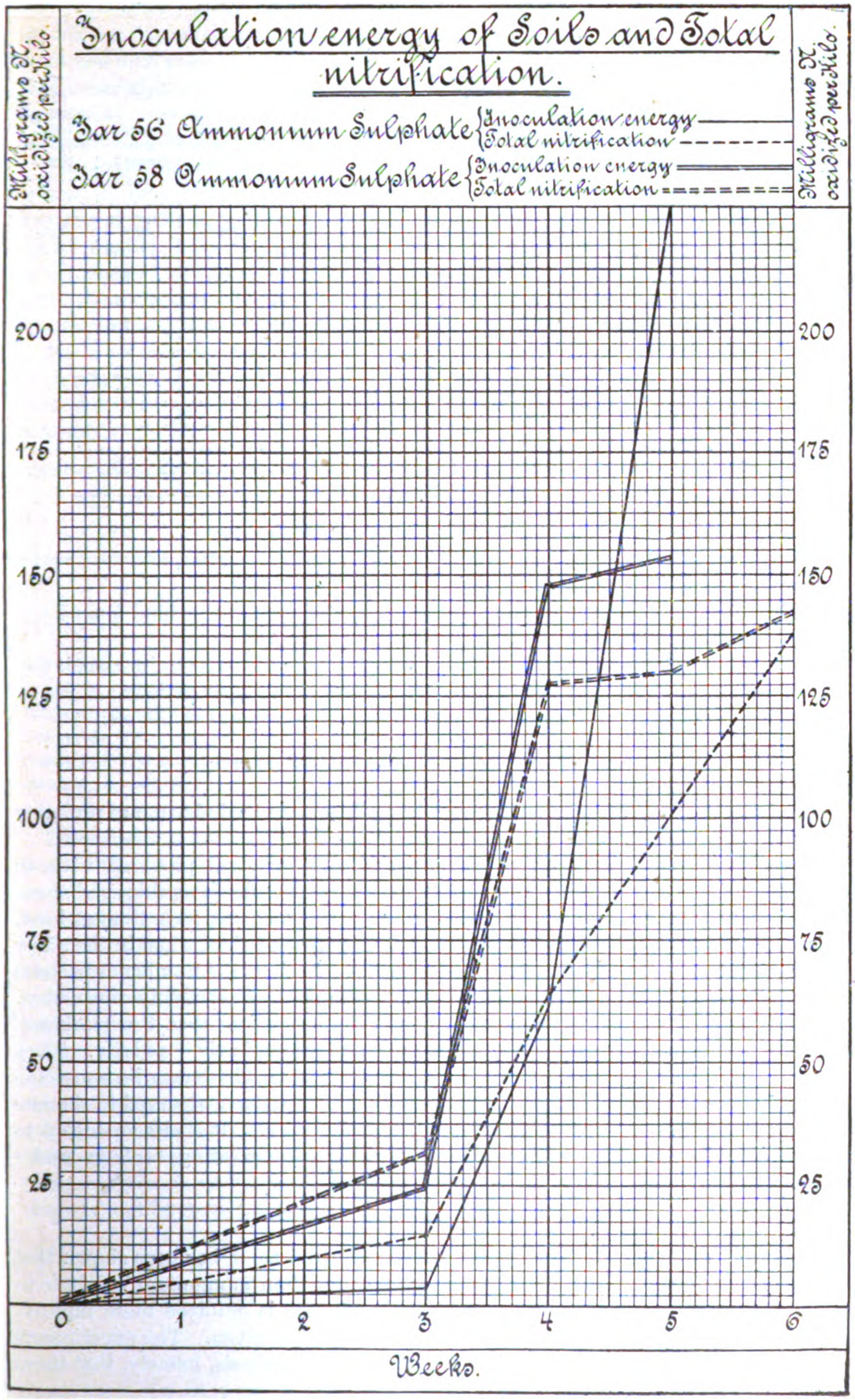

FIG. 5.—Inoculation energy of soils and total nitrification (ammonium sulphate).

TABLE III.—*Effect of time on inoculation energy.*

[Nitrogen oxidized per kilo of soil.]

Nutrient.	Three weeks.		Four weeks.		Five weeks.		Six weeks—total nitrification.
	Total nitrification.	Inoculation energy.	Total nitrification.	Inoculation energy.	Total nitrification.	Inoculation energy.	
Series A, derived from soil No. 1748:	*Mgs.*	*Mgs.*	*Mgs.*	*Mgs.*	*Mgs.*	*Mgs.*	*Mgs.*
Jar 55, cotton-seed meal ...	52	81	193	141	202	205	210
Jar 56, ammonium sulphate	15	4	63	57	....	219	138
Jar 57, cotton-seed meal ...	35	12	144	158	86	97	91
Jar 58, ammonium sulphate	32	25	128	147	133	153	....
Series B, derived from soil No. 1781:							
Jar 91, cotton-seed meal ...	93	115	159	123	....	....	....
Jar 92, cotton-seed meal ...	113	99	171	131	....	....	....
Jar 93, ammonium sulphate	75	60	124	75	....	....	....
Jar 94, ammonium sulphate	24	75	138	139	....	....	....

An examination of the table leads to the following conclusion:

1. The inoculation energy of a soil provided with nitrifiable matter, and kept under favorable conditions, increases with time.
2. The inoculation energy of the soil has a close relation to the total quantity of nitrogen oxidized in the soil.
3. The ratio of the total quantity of nitrogen oxidized in the soil to its inoculation energy decreases from 3:1 or 2:1 in three weeks, to 1:1 in longer periods.
4. This ratio depends to some extent on the inoculation energy of the soil.

CAUSE OF VARIATIONS IN INOCULATION ENERGY.

The changes in the inoculation energy of a soil, as described above, are probably due to the following causes:

1. *Increase in the number of germs present in the soil.*—In a soil provided with an abundance of food for the nitrifying organisms, and kept under favorable conditions, it is self-evident that the number of the bacteria will increase, for a time at least. This increase seems to continue during the whole period of time taken in the study of the changes in the inoculation energy of the soils. In time, the number of bacteria must remain stationary, and then begin to decrease as the food supply diminishes.

2. *Increase in the activity of the organisms.*—At the same time that the number of nitrifying organisms increases, there seems to be an increase in their activity. They either multiply more rapidly, or grow in power to nitrify. There may be some connection between this change and the periodicity which we have observed in the rate of nitrification of ammonium sulphate and cotton-seed meal. It is also analogous to the increase in activity or "virulence" of nitrogen-assimilating bacteria under cultivation.

NATURE OF THE NITRIFYING ORGANISMS.

In previous articles from this station, already referred to, it has been shown that some soils nitrify cotton-seed meal to a greater extent than ammonium sulphate in three weeks, while with other soils ammonium sulphate is oxidized more rapidly. The explanations advanced for this fact have already been given. The experiments now to be described were designed to test the third hypothesis, namely, that there are organisms which convert organic matter directly to nitrites, as well as those which oxidize ammonium salts to nitrites.

In the first place, Is the difference in the rate of nitrification of cotton-seed meal and ammonium sulphate due to the nature of the soil, or to the nature of the organisms in it? The answer to this question is, that the difference is due most largely to the germs, though the nature of the soil is not without its influence.

The experiments which lead to this answer are based on the following considerations: If different samples of a sterile soil are inoculated with the germs from a variety of soils, and allowed to nitrify ammonium sulphate and cotton-seed meal under exactly the same conditions, there will be no difference in the relative amounts of cotton-seed meal and ammonium sulphate nitrified, unless the organisms derived from the different soils are different. That is to say, the soil and other conditions of the experiment do not vary, with the exception of the germs, which do vary.

Table IV contains the results of these experiments. The table shows the number of milligrams of nitrogen in the form of ammonium sulphate, and in the form of cotton-seed meal, oxidized in three weeks in the same sterilized soil inoculated with a number of soils.

TABLE IV.—*Relative amounts of cotton-seed meal and ammonium sulphate nitrified by inoculation of a soil.*

[Nitrogen oxidized per kilo of soil.]

Soil used for inoculation.	Cotton-seed meal.	Ammonium sulphate.	Ratio.
	Mgs.	*Mgs.*	
Loam soil, 1748	73	66	1:0.9
Sandy clay soil, 1746a	100	53	1:0.5
Black sandy soil, 1764a	99	61	1:0.6
Loam soil, 1776a	29	7	1:0.3
Sandy soil, 1772	23	10	1:0.4
Black sandy soil, 1781	100	84	1:0.8

The relative amounts of cotton-seed meal and ammonium sulphate nitrified varies from 1:0.3 to 1:0.9, according to the soil used for the inoculation. This variation is certainly evidence that the variation in the comparative rate of nitrification of ammonium sulphate and cotton-seed meal is due to variations in the organisms in different soils. That is to say, it seems that there are two classes of nitrifying bacteria.

When the nitrification in the inoculated soils is allowed to continue for a longer period of time than three weeks, the relative amount of nitrogen oxidized sometimes varies. Table V exhibits the variation in the relative amounts of nitrogen oxidized in varying times in ammonium sulphate and cotton-seed meal. The sterile soil presents conditions favorable to the development of both classes of organisms, and we might expect both to develop vigorously, but this does not prove that there is no difference in the organisms which effect nitrification in different soils. Even if we exclude the cases of variation, there is sufficient evidence to establish the point that there is a difference.

TABLE V.—*Effect of time on nitrification of cotton-seed meal and ammonium sulphate in inoculated soil.*

[Nitrogen oxidized per kilo of soil.]

Soil No.	Time.	Cotton-seed meal.	Ammonium sulphate.	Ratio.
	Weeks.	*Mgs.*	*Mgs.*	
1746a ..	Five	100	53	1:0.5
	Six	104	156	1:1.5
	Seven	129	187	1:1.5
1768....	Three	3	7	[a]1:2.3
	Four	16	15	1:1
	Five	80	79	1:1
1764....	Four	99	61	1:0.6
	Five	133	74	1:0.6
1766a ..	Three	29	7	1:0.3
	Four	134	68	1:0.5
	Five	435	76	1:0.6
1772....	Three	23	10	1:0.4
	Four	130	70	1:0.5
1781....	Three	100	84	1:0.8
	Four	165	131	1:0.8

[a] Excluded on account of the small amount of nitrification during this period.

Another evidence in favor of the view that there are variations in the nitrifying organisms in soils is offered by a comparison of the relative amounts of cotton-seed meal and ammonium sulphate nitrified in the soil itself, and in the soil inoculated from it. The figures are given in Table VI. It is seen at a glance that, though the ratio of nitrification of ammonium sulphate and cotton-seed meal varies from soil to soil, there is comparatively little variation from a soil to its inoculation This is a further evidence that the variations are due to the germs in the soils.

TABLE VI.—*Relative amounts of cotton-seed meal and ammonium sulphate nitrified in soils and in inoculations from them.*

[Nitrogen oxidized per kilo of soil.]

Soil No.	Method.	Cotton-seed meal.	Ammonium sulphate.	Ratio.
		Mgs.	*Mgs.*	
1748....	Direct	212	176	1:0.8
	Inoculation	73	66	1:0.9
1746....	Direct	176	77	1:0.45
	Inoculation	100	53	1:0.53
1772....	Direct	5	3	1:0.6
	Inoculation	23	12	1:0.5
1781....	Direct	284	267	1:0.9
	Inoculation	105	85	1:0.9

If there are two classes of nitrifying organisms in the soil, it should be possible to increase the relative numbers of one class by the cultivation of it in a soil containing cotton-seed meal, and the relative numbers of the other class by cultivation in a soil containing ammonium sulphate.

If soils containing cotton-seed meal and ammonium sulphate are inoculated from these cultures, the relative amounts of cotton-seed meal and ammonium sulphate nitrified should be different in the two cultures, though they are both derived originally from the same soil. This experiment has been tried and has succeeded, and the results of the work are given in Table VII. In each case the cultures made in cotton-seed meal nitrified a proportionally larger quantity of cotton-seed meal than the cultures made with ammonium sulphate. Also, the cultures made with ammonium sulphate nitrified a larger proportion of the nitrogen in the form of ammonium sulphate than in the form of cotton-seed meal. It is thus possible to increase the relative numbers of the one group of organisms by cultivating them in a soil provided with the appropriate food.

TABLE VII.—*Effect of cultivating the organism on the relative amounts of cotton-seed meal and ammonium sulphate nitrified.*

[Nitrogen oxidized per kilo of soil.]

Conditions.	Cotton-seed meal.	Ammonium sulphate.	Ratio.
Set I (1746):	*Mg.*	*Mg.*	
Direct, 5 weeks	176	76	1:0.5
Inoculation, 5 weeks	135	194	1:1.4
Grown in cotton-seed meal	225	157	....
	240	167	....
Mean	233	162	1:0.7
Grown in ammonium sulphate	190	200	....
	189	....	....
Mean	190	200	1:1.05
Set II (1772):			
Direct	5	3	1:0.6
Inoculation	256	70	1:0.4
Grown in cotton-seed meal	158	34	....
	130	47	....
Mean	143	41	1:0.3
Grown in ammonium sulphate	118	70	....
	114	57	....
Mean	116	64	1:0.6

An attempt might be made to explain the preceding statements on the theory that two groups of organisms take part in the process of nitrification in soils, one group which nitrifies ammonium salts, and one which prepares organic matter for the first group by converting it into ammonium salts. Undoubtedly both classes of organisms of this kind do exist in soils. When the organisms are cultivated in a soil containing ammonium sulphate, the germs which prepare cotton-seed meal for nitrification increase only at the expense of the organic matter in the soil, and so the soil loses in power to nitrify cotton-seed meal and gains in power to nitrify ammonium sulphate. When the organisms are cultivated in a medium containing cotton-seed meal, the organisms which prepare organic matter for nitrification increase, and so the soil gains in power to nitrify cotton-seed meal. But just here the theory fails to explain all the facts. Any increase in the power of a soil to nitrify cotton-seed meal must, according to this theory, be conditioned on an increase in the power or number of the germs which oxidize ammonium salts, since the cotton-seed meal is first converted into ammonium salts. But our experiments show that an increase in the power to nitrify cotton-seed meal is accompanied with a decrease in power to nitrify ammonium sulphate. This fact can not be reconciled with the above theory, but is in favor of the view that soils contain organisms which nitrify organic matter directly. It is very evident that the process of nitrification is a very complex phenomenon, participated in by a number of classes of bacteria.

DETERMINATION OF THE NITRIFYING POWER OF SOILS.

The work recorded in this paper serves as the basis for a method of determining the relative nitrifying power of two or more soils. In order to compare the nitrifying power of two soils, it is necessary that they should be placed under favorable conditions for nitrification, the conditions being exactly alike for both soils. Each soil should contain the same number of organisms of nitrification, with the same degree of activity, and the temperature, water content of the soil, and other conditions should be the same.

The nitrifying power of two soils may be compared in two ways. Of these the second is to be preferred, since it gives more exact results:

Method I.—By comparison of direct nitrification and inoculation tests. Nitrification experiments and inoculation tests are made on the soil as it comes from the field by methods described in the earlier part of this paper. The sterilized soil used for the inoculation tests should be of good quality. If the soil is of good quality, the quantity of ammonium sulphate and cotton-seed meal nitrified in it directly in three weeks should be two to three times the quantity nitrified in the inoculation tests, but if the soil is of poor quality, nitrification will go on more rapidly in the inoculated soil than in the soil itself, although the inoculated soil starts with only one twenty-fifth as many nitrifying germs. The application of the method is illustrated in Table VIII. In two of the soils the ratio of the nitrogen oxidized in the soil and in the inoculation from it is between 2:1 and 3:1, while in the soils which are not such favorable mediums for nitrification the ratio is between 0.4:1 and 0.1:1. In the case of soil No. 1772 a continuation of the experiment for a fourth week shows that it is a much more favorable medium for the nitrification of cotton-seed meal than ammonium sulphate. As stated, the second method now to be described is to be preferred for the determination of the nitrifying power of soils.

TABLE VIII.—*Relative amounts of nitrogen oxidized in soils and in inoculations.*

Conditions.	Direct.	Inoculation.	Ratio.
GOOD SOILS.			
1748, loam soil, with humus:	*Mg per kilo.*	*Mg per kilo.*	
Cotton-seed meal	182	73	2.5:1
Ammonium sulphate	176	67	2.7:1
1781, garden soil, rich in humus:			
Cotton-seed meal	270	98	2.9:1
Do	299	113	2.6:1
Ammonium sulphate	256	75	3.4:1
Do	281	94	3.0:1
POOR SOILS.			
1741, loam soil, little humus:			
Cotton-seed meal	37	102	0.3:1
Ammonium sulphate	20	53	0.4:1
1772, sandy soil:			
Cotton-seed meal	7	21	0.3:1
Do	2	24	0.1:1
Ammonium sulphate	3	8	0.3:1
Do	3	12	0.2:1
1792, 4 weeks:			
Cotton-seed meal	70	156	0.4:1
Do	61	130	0.5:1
Ammonium sulphate	4	70	0.06:1
Do	3	....	....

Method II.—In this method the soils to be tested are compared in their nitrifying power with some standard soil, of good quality, a large supply of which has been secured. All of the soils are sterilized by heating at a moderate temperature for some time, treated with chloroform, and heated again to drive off the chloroform. The heat used in drying the soil should not be too high. Determinations of the water saturation capacity of the soils are made. Inoculation tests are then instituted on the soils as described in the earlier part of the paper. All the soils to be compared should be inoculated at the same time and with the same soil, so that each jar of soil will contain exactly the same number of nitrifying organisms with the same inoculation energy. As the number and inoculation energy of nitrifying organisms in a soil are subject to variations from day to day, it would not do to inoculate some of the soils one day and the remainder the next, but with every set of soils a comparison should be made with the standard soil.

At the end of three weeks a determination of nitrates is made in every sample of soil, and since all conditions are the same, with the exception of the soils, the results are directly comparable, and represent the nitrifying power of the soils. If the soil used for the inoculation has a low inoculation intensity, the time of the experiment should be extended. Table IX shows the application of this method to the determination of the relative nitrifying power of three soils.

TABLE IX.—*Relative nitrifying powers of soils.*

[Nitrogen oxidized per kilo of soil.]

Nutrient.	Poor sandy soil, No. 1779.	Rich sandy soil, No. 1780.	Soil, No. 1747.	Ratio.
	Mgs.	*Mgs.*		
Cotton-seed meal	2	116		
Do	2			
Mean	2	116		1:56
Ammonium sulphate	3	68	71	
Do	3	77	84	
Mean	3	73	77	1:24:26

With these soils the quantity of water used was one-third of their saturation capacity, which of course varies for the different soils. Whether this quantity of water should always be used or not, must be decided by subsequent experiments.

Referring to the table, it is seen that the poor sandy soil has little power of nitrification. The sandy soil used to illustrate Method I has also little power of nitrification, and less for ammonium sulphate than cotton-seed meal. On the other hand, the sandy soil in Table IX, which contains much humus, has a high power of nitrification. These results illustrate the importance of a study of the comparative nitrifying powers of soils.

Mr. EWELL. I would like to ask Mr. Fraps one or two questions. How are the moisture and temperature controlled?

Mr. FRAPS. By weighing the jar containing the soil and replacing the water lost twice a week. The jar was placed in a water bath and kept as nearly as possible at 35° C.

Mr. EWELL. What was the quantity of soil used?

Mr. FRAPS. Five hundred grams. All the details will be found in the paper as published in full.

Mr. VAN SLYKE. I move that we adjourn until 1.30 p. m.

The motion prevailed and the meeting adjourned at 12 o'clock noon.

FRIDAY—AFTERNOON SESSION.

The meeting was called to order by the president at 1.30 p. m.

The PRESIDENT. If there is no objection, we will proceed with the discussion on soils.

Mr. VEITCH. I have been much interested in the question of soil acidity and in the method with which Mr. Hopkins has been working. In the Bureau of Soils we also have been working on methods for the determination of acidity. It seems to me that the first thing we need to do is to define acidity and settle upon an indicator which shall be the standard by which to judge the reaction of soils. Many of the acids that may be present in soils are so weak that they give no certain reaction with most indicators, except in concentrations greater than probably exist in most soil solutions. After a great deal of experimenting with many indicators I have adopted the following method for determining the reaction of a soil:

Ten grams of soil are treated with 100 cc of distilled water in a Jena flask and allowed to stand overnight. Fifty cc of the supernatant liquid are carefully drawn off and boiled with a few drops of phenolphthalein in a covered Jena beaker until the appearance of the pink color or to a volume of about 5 cc with no development of color. The pink color shows the soil to be alkaline, while no color shows it to be acid or neutral.

For the quantitive determination of a soil acidity I use the following method:

To three portions of soil, each consisting of as many grams as the standard limewater contains milligrams of lime (CaO) per cc, add 50 to 60 cc of distilled water and different amounts of standard limewater. For example, to the first add 10 cc, to the second 20 cc, and to the third 30 cc of limewater. Dry down at once on the steam bath, transfer to stoppered Jena flasks with 100 cc of distilled water, allow to stand overnight, with occasional shaking, draw off or filter off 50 cc, place in a Jena beaker, add a few drops of phenolphthalein solution, and boil until the appearance of the pink color, or in case no color is developed, to a volume of about 5 cc. Then with the two portions of treated soils, one of which has been rendered alkaline by the limewater and the other of which is still acid, as guides, prepare three fresh portions of 10 grams each and add limewater as before, except that the amount added to a dish differs from that added to another by only 1 or 2 cc. Dry, take up with 100 cc of water, allow to stand, draw off, and treat exactly as before. The smallest amount of limewater which gives the characteristic pink color is taken as the acidity equivalent of the soil. Each cubic centimeter of standard limewater is equivalent to an acidity of 0.01 per cent expressed as calcium oxid.

The method has been applied to quite a number of soils with very satisfactory results. So far we have had no opportunity to observe the effect on crop production of applications of lime in the amount called for by the method. In quite a number of determinations of the reaction of the soils of clover (red) fields I have never found the acidity of those portions bearing a vigorous crop to exceed 0.0013 gram of calcium oxid per 10 grams of soil, while no clover was found on those portions of the field having an acidity equal to 0.002 gram or more calcium oxid per 10 grams of soil, though these portions may have been only 1 or 2 feet distant from the first-mentioned portions bearing a good growth. These results lead me to hope that the method has an agricultural significance. Work along this line has been planned for the coming year.

Mr. Wiley. I have been much interested in the papers on soils and in the discussion. Of course we all recognize the fact that the soil is the most important subject of agricultural research, in one sense of the word at least, and that the methods which the physicists and chemists devise for the amelioration of the soils are not only of scientific interest but of immense economic importance. The surface of the earth instead of growing larger is growing smaller; therefore, as the area of the earth can not be increased with the increasing population and demand for food unless we have some method for increasing the productivity of the soil, the time will come when the limit to population will be determined by the supply of food. The great question is how to increase the productivity of the soil. One of the important works of agricultural engineering is found in irrigation, by which we may increase the areas which can be used for agricultural purposes. Therefore, it seems that we must look in part to this source to increase the food supply. It is necessary, first, to increase the productivity of the areas which are already under cultivation, and second, to make those areas available which by reason of insufficient rainfall are not tillable. We know what scientific agriculture has done in the way of conserving plant foods and in supplying additional plant foods to the soil. All of the problems which have been presented in these papers are of enormous importance in these investigations. Little was known in a practical way about soil acidity until the investigations of the Rhode Island station brought out the relative acidity of soils. Twelve years ago we studied in Florida absolutely sterile soils, and in the potted soils of that region the plants would make a vigorous start for a few days, but would never reach maturity. Of course the trouble here was that the acidity of the soil prevented nitrification. The investigations at the Rhode Island station showed how fields which were very sterile could be made productive by correcting the acidity of the land. Now we have these investigations to determine the degree of acidity, which we listened to this morning, so that the farmer may not only be able to tell whether his soil is acid, but how much calcium carbonate or lime may be needed to correct this condition.

The distribution of the crops is largely determined by the character of the soil. The old discussion as to whether you can tell anything about a soil by chemical and physical tests has also been brought before us this morning. The soil when left to itself does determine the character of the native crop. The character of the natural growth of the forest is largely determined in any given locality by the nature of the soil itself. I was brought up in a locality where we had almost all kinds of forest trees and the growth of the forest depended largely on the condition of the soil. The best way to study the effect which a soil itself produces upon a crop is to go into the forest and see what it does in the natural state and afterwards study it in an artificial condition.

We know that pine trees grow in the sand, where maple and oak trees will not grow, and, when left to natural causes in the struggle for existence, that plant which is best adapted to the soil and the rest of the environment is the one that survives. It is clear to all, I think, that we should study and know the influence of the soil upon the character and growth of the crop which is to be grown upon it. We all know that the soil which is suitable for growing vegetables is not best adapted to wheat growing. More clay is required for wheat. It is either a question of plant food in the soil or the physical state of the soil which determines this. The physical state of the soil is largely influential in determining these natural growths. We know that oak trees grow on stiff flat soil, while maples and poplars grow in the open porous soils. In southern Indiana, years ago, the people would not try to grow wheat on fields from which oak trees were cleared. They thought wheat would not grow where the oak trees grew—in a flatstiff soil—but that it must be grown on rolling limestone land where the maples grew. Early settlers would first go where the maple trees grew, and you will notice that where the oak trees grew was the last place to be settled. These flat fields are now really more productive than the rolling lands. By adding phosphoric acid to the soil the finest wheat in Indiana is grown on the flat lands to-day.

The soil is useful in proportion to its fertility, granted that the kind of crop that is suitable to the soil is grown. The farmers learned to recognize this fact, and where they formerly produced from 8 to 10 bushels of wheat per acre they are now getting from 30 to 40 bushels per acre.

The magnitude of the crop is determined chiefly by the amount of plant food available in the soil. I have studied this particularly in the case of one or two standard crops, especially in the case of the sugar beet. I find that of all the factors which go to make a rich sugar beet the soil is the least important, but of those which go to make a big crop of beets the soil is the most important. Aside from the principles which I have enumerated, of the natural selection of plants for soils, it is evident that the soil is improved under artificial conditions in proportion as it will produce larger crops, while under these same conditions the character of the crop is determined largely by what we call meteorological conditions. In the case of the sugar beet, granting that the soil will produce a standard crop, say 10 tons per acre, the amount of sugar in the beets depends upon a great many conditions, particularly those of temperature and sunlight. It is necessary that there be sunlight, but not necessarily sunshine. Formerly I said "sunshine," but my investigations have taught me to say "sunlight." The rays which filter through the clouds are almost as useful as if they fell directly upon the plant; in fact in southern latitudes more so as cloudy weather is then an advantageous factor in diminishing the intensity of the heat. This factor is purely a meteorological one. We want plenty of light and an even low temperature to produce good sugar beets. We are studying the effect of meteorological conditions, sunlight, etc., on the gluten in wheat and its other nitrogenous constituents. It is not the soil which determines the amount of gluten or nitrogenous matter in wheat. A long period of growth tends to diminish the nitrogenous content of wheat and increase the starch content. Wheats that are sown in the springtime and harvested within a few months are richer in nitrogen and poorer in starch than wheats planted in the autumn and harvested the next season.

Scientific agriculture, in the progress of agricultural chemistry, is now endeavoring to show the practical farmer what meteorological conditions control the character of his crop, and what soil conditions control the magnitude of his crop, and thus teach him to change its constituents in such a way as to produce those things which are most useful and valuable and in the greatest quantities. In other words, agricultural chemistry, having determined what the soil is, what plants are, and how fertilizers can be made and applied, is now advancing to a still higher plane of investigation to determine how the soil shall be made maximum in production and how the conditions of growth shall secure the maximum of desirable qualities.

The PRESIDENT. If there is no objection, we will take up the subject of sugar. Two papers were presented yesterday. There will be opportunity for discussion.

Mr. WILEY. I would like to say a word of further explanation to some of the members who have adhered to the action of the international committee on sugar analysis. The chairman of this committee, Dr. Herzfeld, has secured by circulars the names of chemists throughout the world who have signified their adhesion to the conclusions of the committee. The purpose is to publish the names from all countries very soon in the Zeitschrift des Vereins der Deutschen Zucker-Industrie, of which Dr. Herzfeld is the editor. I would be glad if the members of this association who are interested in sugar analysis and who have not received that circular would apply for one, so that their names can be inserted in the proof sheets. The object of this publication is to make known those who have adopted the principles which are set forth in the action of this international committee. The committee will meet again in Berlin in June and probably will then be discharged. Of course, we hope to have a large representation from our country, and I think we shall. This report is not a detailed one. It leaves the matter of details to the various associations throughout the world. It has established certain principles in polarization and estimation of sugars. While it may be true that the principles which this committee has adopted are not altogether the best, it is certainly true that they will secure a uniformity of procedure throughout the world.

Other international committees have been appointed for other purposes, and among them is a committee for the establishment of a uniform international method for the analysis of fertilizers. This committee has also reached a general agreement and has adopted as one of the bases of action our own official methods. The adoption of these general principles will go far toward securing uniformity of practice, and a most important step has been taken. An international committee on analytical chemistry has also been appointed, and will hold its first meeting next June, at the time of the assembly of the Fifth International Congress of Applied Chemistry at Berlin. Professor Lunge, of Zurich, Switzerland, is president of the committee. We have on this committee representatives from all parts of the world, making it international.

I must say with some pride that we were the first to attempt to secure uniformity of methods. I believe this whole movement for the unification of methods has sprung from the agricultural chemists of our country. England in most things is a progressive country, but she has made the least progress in this particular, and has but rarely had a representative at the international congresses of applied chemistry. There is one step in the way of uniformity for which the English agricultural chemists deserve credit, that is the adoption of a condenser, modeled after our own, for the determination of volatile acids on dairy products. So even England has taken one step in the direction of uniformity.

The PRESIDENT. I would like to ask if this list of the adherents to the conclusions of the international sugar committee is to be published as a separate report?

Mr. WILEY. I do not think it will be, but we may be able to get separate lists, and when the proof comes of the names of the adherents in this country I will write to Dr. Herzfeld for copies for those who are subscribers, or will endeaver to have the list published in the journal of the American Chemical Society.

The PRESIDENT. In the absence of the referee on tannin, the question of food standards is now before us for discussion. We have the honor to have with us to-day Mr. Hamilton, secretary of agriculture of Pennsylvania, and we shall be glad to hear from him.

REMARKS ON FOOD STANDARDS.

By Mr. John Hamilton,

State Secretary of Agriculture, Pennsylvania.

I was unfortunate in not being here to-day to hear the report which was presented to the association on food standards, and therefore I am not informed as to what has been done. I know that last year certain things were agreed upon, but I have seen no publication of the results. I am not sure whether the committee adheres to the things which were agreed upon then, or whether, in the natural progress of things, changes have been suggested, so that they are still holding these matters in suspense. Those of us who are actively engaged in the work of securing purity of foods for the people in our several States are greatly interested in the work of this association, because the questions that are involved are questions of a scientific character, of a character that can not be determined by the ordinary physician or practitioner in the several States. I am glad to know that this association has taken up the question of food standards in a way that is likely to give the people who have charge of this work an authority which can be quoted in the enforcement of the laws and information that will be reliable. The great difficulty that is encountered by the food officers is that of getting accurate information. Experts disagree with regard to certain facts in our food work, and so when we come into court we find that men of attainments hold certain opinions in regard to the use of food preservatives and coloring matters, while other food experts, graduates of the same school of medicine, are found upon the other side confronting us, before a jury, with their testimony which is exactly contrary to that we have presented. So there is confusion and each case has to be fought out on its own merits, and sympathy for the defendant often determines the action of the jury to the injury of the public. There is need for accurate information that will be accepted and agreed upon by all scientific men with regard to this question of standards, and with regard to the healthfulness or unhealthfulness of food preservatives and coloring matters.

There are three difficulties with which the food authorities have to contend. The first is in obtaining uniformity in methods of analysis. One of the great objects, I suppose, of this association is to secure this uniformity, and it has succeeded in bringing about a great change in this respect, so that substances which are now sent from one chemist to another are likely to correspond in their analyses. But I do not understand that all of the food analysts are entitled to become members of this association, and, therefore, some of the chemists that are now employed in the States are still pursuing methods that have been rejected by this association. Consequently there is discrepancy in their results. If the association could take into its membership food analysts of the several States, it seems to me that it would be a great benefit to those of us who are now trying to enforce the law, for then our analysts would agree in their testimony.

There is another matter of great importance—the question of what shall constitute a standard of purity; how each chemist may know when the substance is of the proper standard. If such a standard was ascertained and adopted for the several States, it would be a great advantage to the manufacturers in their work of preparing food, and there would be a great simplification of the work of the food commissioners in the enforcement of the law. In the State of Pennsylvania we took up this question and did the best we could with it. It was an immediate necessity that some standard should be adopted. We had to meet the question at once and it seemed that we were not likely to get the information from other sources soon, so we called a meeting of all of the food analysts in the State department of agriculture, securing also expert pathologists and others not connected with the department who were interested in the matter of purity of foods, and presented before this meeting the question of standards, and also that of the effects of antiseptics, preservatives, and coloring matter. There were united in that meeting lawyers and scientists, some of the most able men that we have in our State, and we formed a set of rules that we have since used in the prosecution of our work. I will read one or two to give you some idea of what has been done:

1. All foods manufactured, sold, offered, or exposed for sale are held to be represented as pure, unless accompanied by adequate notice to the contrary, in which case they must be distinctly labeled as "mixtures" or "compounds," or as "artificial" preparations.

2. Food sold as pure must be true to name, of standard strength, quality, and purity, and not a compound, mixture, or an artificial preparation or imitation.

3. Where no standard of strength, quality, or purity is fixed by law, the standard required shall be that adopted by the highest recognized authorities, such as the United States Pharmacopœia or the Association of Official Agricultural Chemists.

5. No fraudulent or worthless article, having little or no food value, shall be mixed with standard goods or substituted for them, and be sold as food under the label "compound" or "mixture;" but all foods sold under this designation must be composed of substances recognized as "ordinary articles or ingredients of articles of food."

6. The question of the admissibility of a nonpoisonous or harmless foreign substance in a food may depend upon whether the substance introduced is necessary in order to improve the value or quality of the food, or is fraudulently added as a diluent and cheapener.

7. No food shall be sold under the name of a substance of which it contains none or only an inconsiderable quantity, and when a name is "coined" therefor such name shall not be suggestive of any substance not contained therein.

The wording of the food laws in force in the several States is practically the same. We have held that where the quantity of the component parts of a food or its quality is not fixed by law the standard shall be that adopted by the American Association of Official Agricultural Chemists or by the United States Pharmacopœia. So we look to you for information, and when a case comes into court, and the declaration is made that a certain standard is held by your association, it should be accepted as evidence, the best that could be secured.

We have also another rule, No. 4, that "no food shall have added to it anything that is poisonous or injurious to health," no matter how small the quantity of the poisonous or injurious substance used. The supreme court of our State has sustained us in this position.

We have another rule of importance—I am reading only those which are likely to be especially interesting to you—which permits the "use of a moderate quantity of coloring matter which is not poisonous or injurious to health, provided the goods are otherwise pure and of standard quality."

We were recently confronted with the question as to what we should do in regard to the use of salts of copper in the coloring of vegetables. We have ruled against their use. One purpose of my visit here is to discover what this association holds with regard to the use of these salts; whether they are poisonous and therefore injurious to health or whether they shall be permitted. The question was a practical one with us and we had to answer it at once. We must have evidence that will sustain us. We have always made it a rule in our State to give the public the benefit of the doubt in such cases, when there is any doubt, and to place the responsibility of permitting the use of these doubtful substances upon the courts. It seems to me that a declaration coming from this body on this subject would have great weight. If there is a question as to whether a substance is injurious to health or not, we hold that it is injurious to health, and it remains for those who wish to force it upon the public to show that it is not.

I am glad to know that your committee on food standards is making progress, but it seems to those of us who have to decide upon such questions every day that it is a little slow. If the matter can be hurried a little it will be a great advantage to those who are engaged in enforcing the laws of the several States.

I have also another matter which I wish to call to your attention. I refer to the position we have taken in the matter of the use of preservatives. We are holding that borax, salicylic acid, and other similar substances, such as formaldehyde and benzoic acid, are injurious to health and ought to be prohibited. Food can be preserved without their use and therefore they are wholly unnecessary and should be excluded. I will read our ruling, so as to give you an idea of our position on the question of the use of preservatives:

> 12. Articles of food that can be prepared by the use of improved processes, so as to preserve them from decay or change, shall have no preservative added, other than salt, sirup, sugar, saltpeter, spice, vinegar, or wood smoke

When we have no law that is specific or that gives us authority to designate or fix standards, we hold that if these standards are formulated in a common-sense way the courts will sustain them, and although not statute law they will be interpreted in accordance with the maxim that "the safety of the people is the supreme law." Thus far we have been sustained in the definitions we have made and yet each time we come into court we have to meet the same set of experts, hired by the manufacturers to confront us, and have the same battle to fight over and over.

I think that no more important question is now before us than this question of the proper preparation of food for the consumption of our people. The amount of adulteration is very great. I had occasion in preparing a paper the other day to go over the work of our department for the last two years. Our chemists had analyzed 5,000 samples of food, and over 50 per cent of this food was found to be adulterated in some form, so that the amount of adulteration is still very great, notwithstanding the efforts that have been made to exclude these goods from the markets. I feel that this association ought to disclose, as soon as it safely can, what it has done toward fixing standards and determining the character of preservatives, because the information is so very much needed, and, if we wait until the whole plan is perfected, we shall wait, perhaps, too long, and bring into discredit the work of the food commissioners in the several States, who had to define their position and could not delay.

I want to say to any of you who have ambitions, in a political way, "Don't ever be a food commissioner." That will ruin you. It is the last step that a man takes in political life, if he is honest. A food commissioner needs to have the backing of the best scientific men. If he depends simply on his own information he will have no show in these days. As Secretary Wilson has just stated, one man can not know very many things nowadays; he must get his information from a large number of people, and the best he can do is to find people who know, and then send for them. I have tried to make use of this principle in our department, and accordingly have made use of this association in the preparation of bulletins on a wide range of agricultural subjects. The experiment station work throughout the United States, with which most of you are associated, has done good service to agriculture in general, because it has brought into use a large number of the best, most highly trained scientific minds that the country possesses, for the solution of those difficult questions which embarrass the agriculturist. I am gratified to be here to-day, and am only surprised that the secretaries of the boards of agriculture of all of the States are not here to get information as to what is being done in the line of their work. It certainly is one place to which they can afford to come and get in contact with the men who are out on the firing line of investigation; who are going into the fight and getting facts for daily use. The men who have the administration of agricultural affairs in the several States can not afford to be without this knowledge.

I understand that an appropriation has been made for the investigation of the use of antiseptics, preservatives, and coloring matters in foods. I think this is a most admirable beginning, but it needs to be established upon a broader basis, by the formation of a large-board of experts, who shall give their undivided time to the investigation of these subjects. One man can not do much, but a large number of specialists, united in a board, could take up these subjects, and much information could be obtained in a short time, and time is now of exceeding value, both to the consumer whose health is at stake and to the manufacturer whose money interests are involved.

I appreciate the great work that this association has been doing for agriculture, and the work that it is doing to establish the truth as it relates to the use of various preservatives in the people's food, many of which are certainly injurious to the public health. I am obliged to you for your courtesy in giving me this opportunity of calling your attention to the pressing needs of the food authorities who are intrusted with the enforcement of the laws enacted in the interest of the public health.

Mr. EATON. The National Association of State Dairy and Food Commissioners appointed a committee last year at Buffalo to meet with this association to confer with its committee on food standards to see what aid could be given. The committee consisted of Dr. R. M. Doolittle, of Michigan; Dr. A. S. Mitchell, of Wisconsin; Dr. William Frear, of Pennsylvania, and myself. As I am the only one present, I wish to express my thanks for the courtesy shown me.

The PRESIDENT. I was about to call on Mr. Eaton. We certainly expected to hear from him in regard to this matter. Our constitution provides that analytical chemists connected with the United States Department of Agriculture, or with any State or National institution or body charged with official control of the materials named in section 1, are members of this association.

Mr. VAN SLYKE. I move that the report of the committee on food standards be approved and the committee continued.

This motion was adopted.

The PRESIDENT. If there is no objection we will proceed to the discussion of tannin. Is Mr. Krug present?

Mr. KRUG. Last year we made quite a radical change in our work by entering on a new field. We had been working for a number of years on the analysis of extracts and barks and we concluded that that method was upon a safe basis, and then decided to take up the determination of the total acidity in tan liquors. The determination of the acidity is of considerable importance, because if the liquor becomes too acid it does not make a good leather. Neither does it make good leather when not sufficiently acid. I desire at this time to read the following letter received from Mr. Procter of the International Association of Leather Trades Chemists:

SEPTEMBER 20, 1902.

DEAR DR. WILEY: As I suppose your A. O. A. C. convention will be coming on soon, I send you under another cover a copy of the Leather Trade Review, containing a report of the Leeds meeting of the I. A. L. T. C., instead of waiting for a more official version. You will be pleased to see that we have made a great step toward coming into agreement with the A. O. A. C., your chrome shake method now being permissive, as well as the use of 590 or any other paper. I hope you will see your way to meet us in adopting the same mode of correction for the absorption of tannin in filtration, of which you will have seen fuller details in the Collegium. Of course if you prescribe a definite mode of filtration, there will be no difficulty in fixing also a definite correction applicable to all the ordinary materials, sulphite extracts being generally the only ones requiring a special value, and the whole correction with 590 being a very small one.

As regards our permissive adoption of the A. O. A. C. method, the decision was based on experiments which have not yet been published in full, which showed, that adopting the results of any of the hide powder methods as a standard for that particular method, the results of the filter method with comparatively pure tannins (gallotannic acid, quebracho) could be raised by nearly 100 per cent by the addition of nontanning matters, such as gallic acid, to the solution. Your method gave truer results, but was itself liable to errors of 50 per cent or more, so that it can only be considered as a temporary expedient, and further experiment is eminently desirable to find one less affected by the presence of impurities, and especially of acids. Your method of preparing chromed powder, though all right where large numbers of analyses are made daily, is very troublesome for chemists who only have such work occasionally, and improvements are urgently wanted either in the direction of abridging the necessary washing, or making a powder (either dry or moist) which will keep and act reliably. I am not sure that formaldehyde may not be satisfactorily substituted for chrome in the preparation, and this would much diminish the required washing. We are experimenting in this direction, but do not wish at all to limit others in also taking up the same work. * * *

With kind regards.

Yours, very truly, H. R. Procter.

Mr. **Krug**. With reference to the statements made by Mr. Procter concerning the time lost in preparing chromed hide powder when a chemist makes only a few analyses at a time, I want to say that it is perfectly feasible to make up a considerable quantity at a time, removing as much water as possible and placing it in a tightly stoppered bottle with formaldehyde, when it will keep as long as six months. In regard to the other points which he discusses, I hope that the referee for next year will take up a comparative study of the changes suggested and decide if their adoption is desirable.

Mr. **Wiley**. I have the honor to be a member of the International Association of Leather Trades Chemists. I have, however, attended only one meeting, but hope to attend others. Only European countries are represented. What we want to do, if possible, is to get one of those gentlemen to come over to our meeting and to send Mr. Krug to one of their meetings. There is no doubt of the fact that they want to meet us on common ground. If we send a representative, there would be no difficulty, in my opinion, in securing a perfectly uniform method of analysis, but at the meeting which I attended it was easy to see what the trouble was. There was no one there who had a knowledge of the work except Mr. Procter, who has been president of this association and is an authority on this subject of world-wide reputation. He is thoroughly in earnest in his desire to cooperate with us. I am glad he has approached this association again, and I am sorry that the proceedings which he sent did not reach us. I would suggest on the part of this association that we answer

Mr. Procter's letter and ask him if he can not send a delegate to our meeting or have their delegate confer with ours. I would therefore ask authority to make such a proposition to Mr. Procter in my reply to his letter.

Mr. Krug. I move that such authority be granted.

The motion was adopted.

[Note by the Editor.—In accordance with the above instructions the following letter was sent to Mr. Procter:]

December 12, 1902.

Prof. H. R. Procter,
Leather Industries Laboratories, Yorkshire College, Leeds, England.

Dear Professor Procter: Your letter of September 20, in regard to our official methods on tannin, was read before the meeting of the Association of Official Agricultural Chemists in October. I was instructed by motion at that meeting to make a proposition to you looking toward a conference between a delegate from our association and the International Association of Leather Trades Chemists. We should be glad to have your delegate attend the next meeting of our association, and perhaps it could be arranged to have a delegate from the A. O. A. C. attend your next convention. I shall be glad to receive any suggestions from you, as it would seem that only in some such way can a perfect understanding be reached and any satisfactory degree of uniformity attained.

Very respectfully,
H. W. Wiley,
Secretary A. O. A. C.

The President. The report of the referee on foods and feeding stuffs is now in order.

REPORT ON CATTLE FEEDS.

By C. A. Browne, Jr., *Referee.*

Outline of Work.

The work this year on feeding stuffs was practically along the lines followed by preceding referees. In response to a circular letter inviting cooperation in the work favorable replies were received from eighteen chemists, and to these the following samples were sent:

No. 1. Timothy hay.
No. 2. Distiller's grains.

The sample of hay was drawn from a lot of pure timothy used the past winter for the steer-feeding experiments in the respiration calorimeter at the Pennsylvania experiment station. The distiller's grains were kindly supplied by the H. J. Heinz Company, of Pittsburg, and consisted mostly of the hulls and residues from a corn mash.

The suggestions made by last year's referee, Mr. Krug, were made the basis of the work, which comprised the following:

Determinations of moisture, fat, and pentosans on both samples according to the official method.

Determinations of crude fiber in both samples according to both the official and König methods.

If any time remained for further work after completing the above, it was suggested that a determination of starch be made in the distiller's grains by the diastase method and also a determination of sugars (sucrose and reducing) in the timothy.

In spite of the fact that the early date of this year's meeting shortened the time at the disposal of the reporters and prevented many of them from doing all the work, considerable interest was manifested and reports were obtained from nine chemists.

ESTIMATION OF MOISTURE.

Notable discrepancies exist in the moisture determinations, but these are to be explained partly by differences in the method of drying.

TABLE I.—*Moisture determinations.*

Analyst.	Method of drying.	Timothy hay.	Distiller's grains.
		Per cent.	*Per cent.*
E. B. Holland, Massachusetts	Glycerin and water oven, 100° C	9.14	8.35
T. M. Price, Maryland	Water oven	8.85	8.40
F. W. Woll and G. A. Olson, Wisconsin.	Water oven, 5 hours	9.16	8.40
L. A. Hill, New Hampshire	Water oven, atmosphere of CO_2	9.43	8.85
C. A. Browne, jr., Pennsylvania.	Water oven, 5 hours, 98° C	9.32	8.49
S. D. Averitt, Kentucky	do	9.50	8.75
Do	Caldwell tubes in H, 5 hours, 98° C	9.99	9.24
Do	Caldwell tubes in H, 10 hours, 98° C	10.16	9.42
R. W. Thatcher, Washington	Caldwell tubes in H, 7 hours, 98° C	10.00	9.42
J. H. Norton, U. S. Dept. Agr.	Air bath to constant weight, 7 hours, 100° C	10.35	9.30
Do	Air bath, 5 hours, 100° C	9.72	9.22
Do	Water oven to constant weight	8.98	8.39
Do	Vacuum, 100° C., 5 hours	9.20	9.07
A. W. Bosworth, Rhode Island.	Watch glasses in H, 5 hours, 98° C	8.25	8.37

In the results of Mr. Averitt the maximum loss on drying in air was obtained in five hours, after which there was a slight gain in the case of both substances; by the method of drying in hydrogen the materials also began to increase slightly in weight after ten hours' drying.

The Caldwell method of drying in hydrogen gave very concordant results so far as it was tried. The results obtained by drying in an air bath to constant weight at 100° agree closely with those obtained by the Caldwell tubes. The high results obtained by these two methods clearly show that all the moisture is not removed by the ordinary method of drying in a water oven for five hours. This fact was brought out ten years ago at the ninth annual convention, so that, to quote one of the chemists, "we are thrashing over old straw by opening up this question again."

It does not seem desirable to prescribe one fixed method for the determination of moisture in feeding materials. No method of drying in air at 100° would answer for materials containing easily oxidizable oils, such as linseed meal; in such cases the Caldwell method is to be recommended. On the other hand, the method of drying in hydrogen at 100° C. would not answer for materials containing levulose or invert sugar. In such cases the method of drying in vacuo at 70° C., proposed by Carr and Sanborn[a] in the proceedings of the twelfth annual convention of this association, seems to be the only practicable one. In the choice of a method the chemist must be guided by the nature of the material to be analyzed.

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[a] U. S. Dept. of Agr., Division of Chemistry, Bul. 47, p. 134.

ESTIMATION OF ETHER EXTRACT.

TABLE II.—*Ether-extract determination.*

Analyst.	Timothy hay.		Distiller's grains.	
	Original sample.	Water-free basis.	Original sample.	Water-free basis.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Massachusetts	[a]2.00	[a]2.20	[a]6.21	[a]6.78
T. M. Price, Maryland	1.87	2.05	5.78	6.31
S. D. Averitt, Kentucky	2.01	2.23	6.50	7.17
F. W. Woll and G. A. Olson, Wisconsin	2.02	2.22	6.58	7.18
R. W. Thatcher, Washington	2.44	2.71	6.59	7.27
L. A. Hill, New Hampshire	1.98	2.13	6.20	6.80
A. W. Bosworth, Rhode Island	1.84	2.00	6.41	6.99
M. H. Pingree, Pennsylvania	1.94	2.14	6.13	6.69
Average	2.01	2.21	6.30	6.90

[a] Results, after deducting blank of 0.44 per cent for extract from new capsule, cotton, apparatus, and ether, were 1.56, 1.72, 5.77, and 6.29. It is not customary to make corrections for a blank in determining ether extract, as was done by Mr. Holland, and this seems a point worthy of attention. We may have here an explanation of some of the discrepancies observed between the results of the different chemists.

In a current number of the Chemiker Zeitung[a] Beger calls attention to a deficiency in the ordinary method of fat determination, the point being that the fat is not completely extracted from certain materials, especially such as are high in protein. To obviate this difficulty Beger recommends the use of the pepsin process first advocated by Dormeyer[b] and afterwards studied by Nerking in the analysis of meats. Nerking[c] showed that with dried meats the customary extraction with ether is insufficient to remove all the fat, but that if the material is first subjected to a digestion with pepsin solution the fatty albuminoid compounds are broken up and the fat may then be completely removed by means of ether. Beger has studied this method in its applicability to many feeding materials, and the following figures are taken from his article:

TABLE III.—*Ether extract—digestion with pepsin solution* (*Beger*).

	Rice gluten.	Brewer's grains.	Malt sprouts.	Bran.	Linseed cake.	Cotton-seed meal.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Ether extract, extraction for 12 hours	0.85	8.52	1.82	5.12	9.07	10.02
Ether extract, second extraction for 12 hours	.40	.10	.32	.03	.28	.05
Ether extract after digesting residues with pepsin and again extracting	5.74	1.14	.75	.25	.31	1.09

Results obtained with a number of other materials are also given. In all cases an additional quantity of fat was obtained after digesting the extracted residues with pepsin, the amount in some cases, as with the rice gluten, being very marked. Beger states that digestive coefficients of the fat in rice gluten gave negative values according to the ordinary method of ether extraction, while by the Dormeyer method a positive result was obtained. Physical and chemical tests of the fat extracted after the pepsin treatment showed it to have the same properties as the ordinary fats.

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[a] 1902, **26**, No. 11.

[b] Pflügers, Archiv. für Physiologie, **61**, 1895, p. 341; ibid., **65**, 1896, p. 90.

[c] Pflügers, Archiv. für Phys., **85**, 1901, p. 330.

Your referee has not been able in the limited time at his disposal to give this method a trial; it would seem, though, that we ought to make a study of this method upon a few of our typical feeding materials. The results might show the necessity of revising the digestive coefficients of fat in some of our feeding stuffs. By subjecting the residues from the ordinary ether extraction to the pepsin treatment the two methods can be easily compared without much extra trouble.

The method of treatment is as follows: Three to five grams of substance are digested with one gram of pepsin, Merck (this is found to be free from ether extract), dissolved in 480 cc water and 20 cc of 25 per cent HCl at 37° to 40° C. for twenty-four hours. The residue is then filtered on a Gooch crucible, washed several times with cold distilled water, and then, after drying, extracted with ether in the usual way. The filtrate must also be shaken out with ether several times and the extract from this united with that from the dried residue.

ESTIMATION OF CRUDE FIBER.

At the last meeting of the association Mr. Fraps presented a paper upon the determination of pentosan-free crude fiber by the König method, with the suggestion that the study of this method be taken up by the association. This suggestion was acted upon, and a comparison of the results obtained by the official and König methods upon the two samples sent out is given herewith. The results by both methods exhibit considerable variations and show that the determination of fiber is at present in a most unsatisfactory condition, as indeed it always has been. The results on timothy hay by the official method, especially, show extremely wide and unaccountable variations. There seems to be no determination where the element of personal equation is so pronounced as in that of crude fiber.

TABLE IV.—*Determination of crude fiber.*

Method and analyst.	Timothy hay.		Distiller's grains.	
	Original sample.	Water-free.	Original sample.	Water-free.
OFFICIAL METHOD.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Massachusetts	37.41	41.17	19.93	21.72
	37.40		19.80	
T. M. Price, Maryland	42.10	46.19	13.53	[a] 14.77
S. D. Averitt, Kentucky	35.32	39.04	19.51	21.65
	34.83		19.71	
F. W. Woll and G. A. Olson, Wisconsin	40.70		20.18	
	40.25	44.45	21.48	23.28
	40.20		22.33	
L. A. Hill, New Hampshire	34.77	38.24	18.40	20.20
	34.50		18.41	
R. W. Thatcher, Washington	42.40	46.82	23.64	25.79
	41.87		23.07	
A. W. Bosworth, Rhode Island	36.25	39.55	20.43	22.10
	36.33		20.07	
M. H. Pingree, Pennsylvania	35.44	38.91	19.18	[b] 21.36
	35.12		19.93	
J. H. Norton, U. S. Department of Agriculture	35.14	39.19	19.57	21.57
Average		41.51		22.21

[a] Results omitted from average.
[b] Fiber contains 0.28 per cent N=1.75 per cent protein.

TABLE IV.—*Determination of crude fiber*—Continued.

Method and analyst.	Timothy hay.		Distiller's grains.	
	Original sample.	Water-free.	Original sample.	Water-free.
KÖNIG METHOD.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Massachusetts	31.69	34.44	21.49	23.27
	30.90	....	21.17	....
T. M. Price, Maryland	21.15	....	....	....
	24.83	[a]22.73	....	....
	16.18	....	....	....
S. D. Averitt, Kentucky	25,20	[a]26.32	14.60	[a]17.23
	22.10	....	16.61	....
F. W. Woll and G. A. Olson, Wisconsin	32.00	....	24.31	....
	33.10	35.85	23.51	26.08
	32.62	....	23.85	....
L. A. Hill, New Hampshire	31.68	35.11	20.32	22.11
	31.91	....	20.00	....
R. W. Thatcher, Washington	32.94	36.79	25.01	27.61
	33.27	....	....	....
A. W. Bosworth, Rhode Island	32.83	35.78	22.11	24.22
	32.83	....	22.27	....
C. A. Browne, jr., Pennsylvania	[b]31.68	34.89	[c]22.24	[d]24.60
	31.60	....	22.81	....
J. H. Norton, U. S. Department of Agriculture	31.15	34.75	21.21	23.38
Average	....	35.37	....	24.47

[a] Results omitted from average.
[b] Fiber after boiling with 1.25 per cent NaOH is 27.49 per cent.
[c] Fiber after boiling with 1.25 per cent NaOH is 17.78 per cent.
[d] Fiber contains 1.09 per cent N=6.81 per cent protein.

COMMENTS OF ANALYSTS.

This being the first general trial of the König method, it is thought well to give the comments of the different analysts in full:

E. B. Holland.—Considerable difficulty was experienced in the filtration, linen being used for the purpose. The final product, even after prolonged washings with hot water, alcohol, and ether, was not as clean as desired.

T. M. Price.—König's method for crude fiber I did not find satisfactory, as it was impossible to get concordant results with duplicates.

S. D. Averitt.—Much difficulty was had in filtering the fiber obtained by the König method. It was necessary to dilute the liquid with water and allow the fiber to settle before it could be filtered, and even then the process was very slow. If the filtration is always as difficult as we found it, the method can hardly be considered practical.

F. W. Woll.—The filtration by the König method took from 1 to 10 hours. To help along the filtration in one determination, the mixture was diluted with hot distilled water after boiling, and the filtration then took only 8 minutes.

R. W. Thatcher.—König's method for crude fiber is a very satisfactory one as far as ease of manipulation and agreement of duplicates is concerned. It gives results that are in no way comparable with those obtained by the official method, however. Some difficulty was experienced in filtering in the determinations made on the distiller's grains, the first sample being ruined in the attempt. In the case of the second sample the method of filtration outlined in the accompanying note (p. 154) was used with very satisfactory results, although filtration was slow even with this method of procedure, the material being not wholly removed from contact with the glycerol-sulphuric acid for nearly an hour after the filtration was begun. This is not a serious objection to the König process as compared with the official method, however, since in the latter even more difficulty was experienced in filtering off the alkaline extract.

A. W. Bosworth.—In the König method for crude fiber, after digestion with the glycerol-sulphuric acid, I had a great deal of trouble in filtering. The timothy hay took between six and seven hours to filter and wash.

J. H. Norton.—Fiber by the König method was filtered in a funnel with a 2-inch porcelain plate and asbestos, no platinum being available. The timothy hay gave trouble, as it was so loose as to allow it to suck through with some asbestos. In each case the washing was continued until the liquid came through clear. Attempt was made to filter the distiller's grains the same as the hay, but the filter clogged and had to be transferred to a 3-inch Hirsch funnel with asbestos. Alcohol seemed to have a greater solvent effect than ether and alcohol. The fiber from the distiller's grains gave trouble in burning on account of the large quantity of asbestos it was necessary to use.

COMMENTS OF REFEREE.

The claim that the König method is shorter than the official method does not seem to be realized by all the chemists, the complaint of the difficulties in filtering, where the König process was used, being very general. Your referee has found the use of a flat-bottomed perforated porcelain funnel, the kind designated in German laboratories as a "Nutsche," and listed in our catalogues as a Büchner funnel, very convenient for difficult filtrations. By using a filter of linen or of hardened paper and by first diluting the hot glycerol-acid mixture with boiling water, the filtration and washing by the König method, employing such a funnel as the one just described, occupied not over ten minutes. The most serious objection to the König method, as it now stands, in the opinion of your referee, consists not in the difficulty of filtration, for that may be in a great measure prevented, but in the danger we run of obtaining an impure quality of fiber. The fiber is, to be sure, practically free from pentosans, and a distillation with hydrochloric acid gives only traces of furfurol, but the boiling glycerol-sulphuric acid mixture does not effect, on the other hand, anywhere near a complete removal of the albuminoid matter in materials high in protein. It will be noted in Table IV that a lower percentage of fiber was found by the König method with the hay, while with the distiller's grains the König method gave in most cases several per cent more fiber. This is undoubtedly due to the high percentage of protein left in the fiber obtained by König's process, this containing in one case 1.08 per cent nitrogen, equivalent to 6.81 per cent protein. The official method also does not give us a fiber absolutely free from albuminoids; it is, however, much purer in this respect than the fiber obtained by the König process, the amount found in the fiber from the distiller's grains by the official method in one case being 1.75 per cent, about one-fourth that obtained by the König method.

Your referee believes we would experience this difficulty with the König method on all feeding materials high in protein, so that we would have little to gain by adopting a method which, while securing greater accuracy on one side, incurs still greater errors on another. As Mr. Thatcher observes, the two methods give results that are in no way comparable. The difficulty may, perhaps, be obviated by modifying the method of König, so that it will include a treatment of boiling the fiber with alkali as with the official method. Your referee has made determinations of the fiber in the two samples, using such a modification of the König method, and obtained 27.49 per cent fiber for the timothy hay and 17.78 per cent for the distiller's grains, results several per cent lower than those obtained by the simple boiling with the glycerol-sulphuric acid mixture. The resultant fiber was much whiter after boiling with the alkali; it contained 1.45 per cent protein and seemed to be nearly free from lignin, as well as from pentosans.

Your referee would recommend a further study of the König method for next year, with the additional treatment of the fiber with alkali as by the official method. We may be able in this way to arrive at a better method of estimating fiber. It does not seem right to throw the matter up without further trial. The results on timothy hay show that seven chemists obtained results by the König method agreeing within nearly 2 per cent, something that can not be said of the official method. Your referee does not believe that the König method as it now stands should be adopted by the association.

ESTIMATION OF PENTOSANS.

The results on pentosans, so far as reported, are presented herewith:

TABLE V.—*Determination of pentosans.*

Analyst.	Timothy hay.		Distiller's grains.	
	Original material.	Water-free basis.	Original material.	Water-free basis.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Massachusetts	23.69	26.23	21.24	23.18
L. A. Hill, New Hampshire	21.94	24.22	21.95	24.08
A. W. Bosworth, Rhode Island	24.71	26.93	22.07	24.09
C. A. Browne, jr., Pennsylvania	24.61	27.14	21.99	24.01
J. H. Norton, U. S. Department of Agriculture	23.58	26.30	21.00	23.15
Average	23.71	26.16	21.65	23.70

By way of comment, Mr. E. B. Holland says: "Not being in favor of the new suggestion as to the amount to be taken, 3 grams were used as usual. The phloroglucol used was Mercks chemically pure, and the solution was prepared in the proportion of 6.5 grams to 750 cc of 12 per cent hydrochloric acid."

Your referee would like to recommend at this point the adoption by the association of the new factors of Kröber for calculating pentosans.[a] The factors at present in use, worked out originally by Tollens and Krüger, are now really obsolete and should be discarded. As is stated in an abstract[b] in the last number of the Experiment Station Record[c] "the close concordance between the data given by Rimbach and Kröber indicates the correctness of the new figures." One principal objection to the present factors is that they can not be successfully applied to small amounts of phloroglucid, the subtraction of the figure 0.0104 from the calculated weight of furfurol frequently giving negative results.

Considerable study is being devoted to the subject of pentoses and pentosans in Germany just at present, as is seen by the numerous contributions of Tollens, Salkowski, Neuberg, and others. The recent discovery by Hammarsten of pentosans in the nucleo-proteids of the pancreas and in other parts of the animal organism has especially stimulated researches in this direction, more however, from the physiological side. Good workable methods have been devised for the qualitative separation of the pentoses from one another, use being made of the various substituted hydrazins, such as benzylphenyl hydrazin and diphenyl hydrazin, and successful attempts have been made toward using these reagents quantitatively. Your referee has employed the benzylphenyl hydrazin with success in isolating arabinose from different materials, and has a specimen of the arabinose benzylphenyl hydrazone which he has recently obtained from timothy hay. There is no reason why these and other newly discovered methods may not be successfully employed to increase our knowledge of the constitution of many feeding materials.

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[a] Journal für Landwirthchaft, 48, 1900, p. 375–384.

[b] For fuller particulars concerning Kröber's factors see the original articles by Kröber, Journal für Landwirthschaft, 1900, p. 375; 1901, p. 7. Also articles by Tollens, Zeitschrift für angewandte Chemie, 1902, pp. 477 and 508, and Zeitschrift für physiologische Chemie, 1902, p. 239.

[c] Vol. 14, No. 1, p. 9.

ESTIMATION OF STARCH.

Only two chemists made reports upon starch, the others finding it impossible to do this part of the work from lack of time. The following results were obtained:

TABLE VI.—*Determination of starch, diastase method.*

Analyst.	Distillery grains.	
	Original material.	Water-free basis.
	Per cent.	*Per cent.*
E. B. Holland, Massachusetts	4.95	5.40
C. A. Browne, jr., Pennsylvania	2.17	2.37
Average	3.56	3.89

The distillery grains had the appearance of being well malted, and the microscope showed with iodin only traces of starch. In a similar sample of distiller's grains which your referee examined some years ago 2.66 per cent of starch was found; the pentosans were also determined just before and just after the malt digestion. Pentosans in residue before malt digestion were 22.16 per cent of the original substance, and after, 20.98 per cent; the difference, or pentosans removed by malt digestion, being 1.18 per cent.

Similar results are reported by Lindsey[a] upon corn meal, wheat middlings, and lupine seeds. There would therefore appear to be a danger that with materials high in pentosans small amounts of the latter may be dissolved during the malting process, being afterwards hydrolyzed and estimated as starch. A study of this point was made some yeats ago by the association, by determining the pentosans in the extracts after the malt digestion. This never seemed to show very much, principally for the reason, already indicated. that our present formulæ are not adapted to calculating small amounts of pentoses or pentosans. Applying Kröber's formula to the weights of phloroglucid would show an appreciable amount of pentosans in these extracts.

As an illustration of this, a quantity of xylose having been obtained from timothy hay, 10 mg (1 per cent of 1 gram) were distilled with hydrochloric acid and precipitated with phloroglucin as usual. The weight of phloroglucid obtained was 10.3 mgs. This corresponds to 14 mgs xylose (1.4 per cent of 1 gram), using Kröber's formula. The formula now employed gives a minus quantity.

To study better the effect of diastase upon the pentosans, the experiment was tried of first isolating the pentosans from other substances before beginning the digestion with malt. Several hundred grams of timothy hay were finely ground, washed first with cold dilute hydrochloric acid, then with water, and lastly with boiling alcohol. From the hay thus purified the pentosans were extracted with dilute soda, precipitated with alcohol, then purified and dried according to the method of Wheeler and Tollens.

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[a] U. S. Dept. Agr., Division of Chemistry, Bul. 51, p. 91.

Working upon this material the following results were obtained:

	Per cent.
Pentosans removed by malt digestion (by inverting and determining copper reducing power)	[a] 28.56
Pentosans removed by water digestion alone (by inverting and determining copper reducing power)	[a] 21.42
Difference, or pentosans removed through diastase	7.14

The experiments were carried out together under exactly similar conditions. The time of digestion was six hours. The results would indicate that diastase seems to exert some solvent effect upon the pentosans, at least when isolated. This solvent action is, however, undoubtedly much diminished when the pentosans exist in combination with cellulose and lignin in cellular tissues. The error introduced into the starch determination with our common feeding materials, judging from the percentage of pentosans removed during the malt digestion, would probably rarely exceed 1 per cent.

RECOMMENDATIONS.

It is recommended that after the sixteenth line the method for determining pentosans be changed to read as follows, following the directions prescribed by Kröber:

> The solution is made up to 400 cc with 12 per cent hydrochloric acid and allowed to stand overnight. The amorphous black precipitate is filtered into a tared Gooch through an asbestos felt, washed carefully with 150 cc of water in such a way that the water is not entirely removed from the crucible until the very last, then dried to constant weight by heating four hours at 100° C., cooled, and weighed in a weighing bottle, the increase in weight being reckoned as phloroglucid. To calculate furfurol, pentose, or pentosan from the phloroglucid, use the table by Kröber.[b]

Mr. BROWNE. I have a communication on filtration in determinations of crude fiber from Mr. Thatcher, which he authorizes me to read.

NOTE ON FILTRATION IN DETERMINATIONS OF CRUDE FIBER.

By R. W. THATCHER.

All analysts of food products, or feeding stuffs, are familiar with the difficulty which is often experienced in filtering off the acid and the alkaline liquids in the determinations of crude fiber by the official method of the Association of Official Agricultural Chemists. Hence I wish to suggest the following modification of the usual procedure, which I have found to give very satisfactory results:

Select a funnel of sufficient size to contain the entire bulk of the mixture to be filtered and fit into its point a small platinum filtering cone. Introduce enough ignited asbestos wool to fill the cone a little more than full. Upon moistening, the wool softens into a fluffy mass, which may be drawn down into a firm, close filter by suction. The mixture to be filtered is now poured into the funnel, with the usual care to avoid disturbing the asbestos layer, and suction is applied. The filtrate obtained in this manner has always been found to be free of suspended particles of fiber. Filtration is very rapid in all cases except when working with finely ground flour or spices, which tend to clog the filter and impede the flow of the filtrate. In such cases place the funnel in an ordinary jacket of boiling water or steam in order to secure hot filtration, transfer the entire mass to be filtered to the funnel, and apply the suction as usual. Filtration will then proceed at the desired temperature and without further attention from the operator, thus avoiding the two objections to the use of the Gooch filter as recommended in the official method. After filtration and washing are complete, transfer the contents of the funnel to a platinum evaporating dish, rinsing the last particles of fiber from the funnel to the dish with a fine jet of distilled water. Evaporate off the water thus used, dry to constant weight, and complete the determination as usual. I prefer this mode of operation to the use of a paper filter, with correction for loss of weight sustained by the paper in a blank determination, as suggested by Winton,[a] for the reason that, in addition to the possibility of obtaining additional fiber from the paper used in the acid filtration, I have found that duplicate determinations of the correction to be applied do not always give concordant results, probably because of variations in the weight and composition of individual filters in any given pack of them. I have used the method outlined above for several years and upon a great variety of samples with uniformly satisfactory results. In the cases where filtration is slow it proceeds without attention from the analyst, thereby relieving the tediousness of the operation very materially.

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[a] Pentosans = dextrose × 0.97 × 0.88. Stone Am. Chem. Jour., **13**, 73.

[b] See report of chairman of Committee B for factors. For table in full see "Journal für Landwirthschaft, **48**: 1900, pp. 375–384.

Mr. Wiley. I have a paper from Mr. Parsons on foods and feeding stuffs, which I think should be presented at this time. Mr. Parsons regrets that he is not here to attend the meeting, and offers some suggestions in regard to the analysis of malt liquors:

SUGGESTIONS AS TO THE ANALYSIS OF MALT LIQUORS.

By Charles L. Parsons.

I have recently had occasion to make analyses of a large number of samples of beer in connection with legal work in this State where the question is not so much to determine the alcohol content as to prove the malt origin of the liquor under question. This makes a very complete analysis necessary, and I think that several important changes can be advantageously made in the methods which will insure much greater rapidity without sacrificing accuracy. I beg to submit the following suggestions:

Alcohol.

Discard the use of hydrometer altogether. The best made are not sufficiently accurate. Use the picnometer with thermometer stopper, and allow the use of any temperature from 15.6 to 25°C. according to temperature of atmosphere, the picnometer to be calibrated with distilled water at the same temperature. This may seem a radical departure and will be criticised, but a study of the coefficient of expansion of pure water and mixtures of alcohol and water[b] will show that with such dilute solutions of alcohol as are under consideration the error would be less than 0.01 per cent. This error is less than would result from evaporation due to capillarity around the ground-glass stopper if the picnometer is filled at 15.6°C. and then brought to room temperature to avoid condensation on the glass, is much less than would arise from the use of a hydrometer, and is fully as accurate as by calculation from the tables of Squibb.

Protein.

Boil down the beer taken to a small bulk, but not to dryness, after acidification with a few drops of sulphuric acid before the main quantity of sulphuric acid or potassium sulphate is added. This will do away with the foaming which is otherwise almost uncontrollable.

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[a] Conn. Agr. Expt. Sta. Report, 1898, p. 189, and Bul. 65, Bur. of Chem., U. S. Dept. Agri., pp. 58, 154, and 155.

[b] Makins, Jour. Chem. Soc., 99: 224, and Silbermann, Comptes Rendus, October, 1848.

PHOSPHORIC ACID.

The uranium acetate method, which is so extremely rapid, can be applied with accuracy to the beer ash if the ash is first moistened with concentrated hydrochloric acid and the acid evaporated in the water bath. I have made comparative analyses with the gravimetric molybdenum method and find that, if expressed in the customary terms of percentages of the beer taken, the method can be depended upon to the third decimal place with certainty. The end point of the titration is exceedingly sharp when the ash is used. If acetic acid is substituted for hydrochloric, or if, after evaporation of the acid the ash is heated much above the temperature of boiling water, part of the phosphates are not attacked by the uranium solution. Use 50 cc beer for the ash and a uranium acetate solution, one cc of which is equivalent to 0.01 per cent phosphoric acid.

SULPHATES IN ASH.

Incorporate a method for the determination of sulphates and allow the use of the regular gravimetric method and the photometric method of Hinds;[a] this latter can be applied to the same solution as is used to determine phosphoric acid after it has been acidified with hydrochloric acid. Hinds's own tables agree much more closely with the gravimetric than those of Jackson.[b] By using the uranium method for phosphates and the photometric for sulphates, these two determinations can be completed in about fifteen minutes after weighing the ash.

ASH.

Use 50 cc of beer instead of 25 cc in order to correspond with the determinations of phosphoric acid and sulphates.

FREE ACID.

Dilute 10 cc of the beer several times with distilled water, add phenolphthalein, and titrate with tenth normal sodium hydrate. The end point is sharp and agrees with that obtained by delicate litmus paper.

Mr. WILEY. I have just received a letter from Mr. Shutt, who regrets his inability to be present on account of an attack of appendicitis. He had hoped up to the last moment to come and fears by reason of his indisposition that he will not be able to serve as referee, but hopes to remain the associate, as at present.

The PRESIDENT. Is it the pleasure of the association to communicate through our secretary the best wishes of the association to Mr. Shutt?

Mr. BOSWORTH. I move that the secretary be instructed to communicate our best wishes to Mr. Shutt.

The motion was adopted.

[NOTE BY THE EDITOR.—The following letter was sent to Mr. Shutt in accordanc with the above instructions:]

OCTOBER 9, 1902.

Mr. F. T. SHUTT,
Experimental Farm, Ottawa, Canada.

DEAR SIR: Upon the receipt of the unwelcome news of your illness, at the last annual meeting of the Association of Official Agricultural Chemists, I was instructed to extend to you the sympathy of that body, together with their regrets that you should be absent from the meeting, and their earnest wish for a speedy recovery. To this I would add an expression of my own personal regret and the hope that you are now on the road to recovery.

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[a] Jour. Amer. Chem. Soc., 22: 269. [b] Ibid., 23: 799.

Sincerely,

H. W. Wiley, *Secretary A. O. A. C.*

Mr. **Van Slyke**. I move that we adjourn until 9.30 to-morrow morning.

The motion prevailed, and the meeting adjourned at 4 o'clock p. m.

THIRD DAY.

SATURDAY—MORNING SESSION.

The meeting was called to order by the president at 9.30 a. m.

REPORT ON INSECTICIDES AND FUNGICIDES.

By J. K. Haywood, *Referee.*

The work for this year upon insecticides and fungicides has consisted principally in testing various methods that have been gathered together in previous years.

Outline of Work.

The following circular letter was sent out to all the station chemists:

> **Dear Sir**: Having been appointed referee on insecticides and fungicides by the Association of Official Agricultural Chemists, I desire to know if you will cooperate with Mr. Emery, the associate referee, and myself in the work.
>
> The subject will be divided into six groups, as follows:
>
> 1. Potash and soda lyes and soaps.
> 2. Arsenical insecticides.
> 3. Copper in copper carbonate.
> 4. Cyanogen in potassium cyanid.
> 5. Formaldehyde in formalin.
> 6. Nicotin in tobacco and tobacco extracts.
>
> Will you kindly let me know what group or groups you will be able to work upon, and oblige,
>
> Yours, very truly, J. K. Haywood, *Referee.*

It was hoped that a seventh class, petroleum products, could be added, but the chemist who was to work upon this class has not reported any results.

Twenty-three replies were received to this letter, 15 favorable and 8 unfavorable. Including the referee, 16 signified their desire to cooperate in the work. Thirteen asked for samples of arsenicals, 7 for samples of copper carbonate, 5 for samples of potash and soda lyes and soaps, 5 for samples of formalin, and 5 for samples of tobacco extract.

Samples and copies of the methods of analysis[a] were forwarded to those who desired to cooperate, with the request that they first perform the analysis exactly as directed, then suggest any changes that they might think necessary to either expedite or render more accurate the method as outlined.

The following reports have been received: Nine on arsenicals, 4 on copper carbonates, 4 on potassium cyanid, 4 on soda lye, 2 on whale-oil soap, 5 on formalin, and 3 on tobacco extracts.

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[a] See Circular No. 10, Bur. of Chem., U. S. Dept. of Agr., "Methods for the analysis of insecticides and fungicides."

Paris Green.

The following table shows the comparative results obtained on Paris green by employing the methods of analysis referred to above:

Table I.—*Results of work done on Paris green.*

Analyst.	H_2O.	Total As_2O_3.		Soluble As_2O_3.		Soluble CuO. Method II.	Total copper oxid.	
		Method I.	Method II.	Method I.	Method II.		Method I.	Method II.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
L. H. Smith, Illinois	0.75	57.75	56.65	0.78	3.10	0.95	29.83	31.43
	.68	57.63	56.65	.78	2.85	.70	29.83	31.30
	....	57.75	....	....	....	....	....	31.43
	....	57.50	....	....	....	....	....	31.43
	....	....	....	....	....	....	....	31.43
H. T. Beans, Idaho	.60	56.82	56.44	.38	2.06	.64	30.00	28.88
	.63	56.89	56.45	.41	2.08	.40	30.12	29.32
	....	56.70	56.45	....	....	....	30.08	29.45
	....	56.89	....	....	....	....	30.24	....
T. M. Price, Maryland	.61	56.75	57.16	.60	2.72	3.45	30.31	30.15
R. W. Thatcher, Washington	.41	57.08	56.21	.62	2.77	1.05	30.80	30.78
	.43	57.15	56.19	.65	2.22	.35	30.56	30.45
D. L. Cleaves, Massachusetts	.37	56.78	54.83	1.67	....	....	....	30.71
(Hatch).	.40	57.02	54.83	1.63	....	....	....	30.40
	....	....	....	....	....	....	....	30.52
R. J. Davidson, Virginia	.63	56.73	56.61	.53	3.63	1.05	30.60	31.19
	.63	56.78	56.56	.54	3.63	1.05	30.60	31.40
	....	56.76	....	....	3.63	....	30.60	31.19
	....	56.74	....	....	....	....	30.32	31.40
	....	56.74	....	....	....	....	30.32	....
	....	56.74	....	....	....	....	30.32	....
H. J. Warner, Bureau of Chemistry,	.53	57.21	57.09	.73	3.87	....	30.42	[a]30.97
U. S. Department of Agriculture.	....	57.09	....	.77	....	....	....	30.97
James Emery, Biochemic Division,	.59	56.66	56.36	.95	....	....	30.10	30.45
Bureau of Animal Industry, U. S.	.57	56.66	56.66	1.00	....	....	30.35	30.45
Department of Agriculture.	....	....	....	....	....	....	....	30.78
J. K. Haywood, Bureau of Chemistry, U. S. Department of Agri-	.73	56.93	57.39	.74	2.94	.50	30.43	[b]30.73
istry, U. S. Department of Agri-	.60	57.16	57.04	.74	2.85	.50	30.43	30.73
culture.	....	57.16	57.17	....	....	....	....	31.17

[a] A determination of copper oxid by weighing the oxid gave 31.07 per cent.

[b] Determination of copper by electrolysis gave 30.87 per cent copper oxid.

COMMENTS BY ANALYSTS.

L. H. Smith.—In the determination of soluble copper oxid the precipitate was not burned with powdered sulphur.

H. T. Beans.—In Method II, for total arsenious oxid, it is much shorter and just as accurate to standardize the iodin solution against a solution of arsenious oxid made by dissolving chemically pure arsenious oxid in a hot solution of sodium bicarbonate.

D. L. Cleaves.—In the determination of total copper oxid by Method I, no matter how much sodium thiosulphate was added, no end point could be obtained. In the determination of total arsenious oxid by Method II it was impossible to obtain a clear solution with hydrochloric acid.

R. W. Thatcher.—Method II, for total arsenious oxid, is in my opinion a very excellent one for work with samples of pure green, but unfortunately the cold dilute hydrochloric acid used in dissolving the green does not completely dissolve any crystals of free arsenious oxid that may have been added. My experience is that such adulteration is by no means so uncommon as to warrant the adoption of a method which will fail to include free arsenious oxid in the total as determined by it.

The following comparative determinations of the total arsenious oxid in some commercial samples of paris green received for analysis at this laboratory were made at the same time that the above work was done:

TABLE II.—*Comparative determinations of total arsenious oxid.*

Sample.	Total arsenious oxid.		Soluble arsenious oxid, Method I.
	Method I.	Method II.	
	Per cent.	*Per cent.*	*Per cent.*
1	56.32	56.33	0.68
2	56.60	56.58	.70
3	56.71	56.20	.84
4	61.25	52.23	10.89
5	62.24	50.37	12.16

DISCUSSION OF RESULTS.

The figures in Table I for the amount of arsenious oxid by Method I show that the agreement between the figures obtained by each analyst and the agreement between the work of different analysts are most excellent. In fact, there are few of our methods which would give better results in the hands of different chemists working upon substances containing such large percentages of the component to be determined. It is true that one set of results is slightly higher than the rest, but they are very little higher, and this could very easily arise from a difference of 0.2 cc in the burette reading.

In the third column are given the results obtained by Method II for total arsenious oxid. It will be seen at once that these figures, obtained by different analysts, agree very nearly as well as those obtained by the previous method, with one exception. The general tendency, however, seems to be in the direction of obtaining slightly lower figures by this method than by Method I. This is doubtless due to the fact that the free arsenious oxid present in the Paris green does not go into solution with the dilute hydrochloric acid used. The referee has observed many times that quite long stirring is necessary to cause the free arsenious oxid to go into solution when pure samples of Paris green are used, and that it is practically impossible to get the free arsenious oxid in solution when samples containing large amounts of free arsenious oxid are used. In this connection it will be noticed that the experience of other analysts has been the same as that of the referee and that Mr. Thatcher gives figures to prove the truth of this statement. I would also add that, as Mr. Beans says, the method can be shortened by standardizing the iodin directly against chemically pure arsenious oxid that has been dissolved in sodium bicarbonate. This, unlike sodium hydroxid, does not seem to cause the arsenites to be oxidized to arsenates in boiling solution.

Neither of the methods for soluble arsenious oxid give very accurate results. In Method I, which is supposed to show the free arsenious oxid existing as such in the green, excluding the extremely high figures 1.67 and 1.63, the results vary from an average of 0.40 per cent to an average of 0.98 per cent. While this showing would be very poor in most determinations, it is not so unsatisfactory in this particular case as it is only to gain an approximate idea of the amount of the arsenious oxid existing as such in the Paris green that this determination is made. In fact, we do not at the present time know within half a per cent how much free arsenious oxid different plants can stand. Method II, leaving out the extremely high figure 3.87, gives results that vary from an average of 2.07 per cent to an average of 3.63 per cent. The figures obtained by this method seem to the referee to give some idea of the stability of the green, and hence this determination is a rough measure of the action of the insecticide in actual orchard practice. Looking at the matter in this light, it is seen that if the results by Method I are low, and by Method II high, the green is unstable and consequently unfit for use, while if the results by both these methods are high, then free arsenious oxid is present as such in the green, thus again rendering it unfit for use. The only way that results of a comparative nature can be obtained by Method II is to follow extremely strict rules as to the amount of water to be used, the length of time of extraction, and the number of times per day that the mixture of green and water is to be shaken.

The figures for soluble copper oxid have no special significance, but the determination was made in the hopes that someone would, during the year, work out (if such a thing is possible) the relation between the green broken up, and the soluble copper oxid that goes into solution.

The averages of the results obtained by different chemists following Method I for total copper oxid agree fairly well with each other. Only one chemist seemed to experience any difficulty in carrying out this method. Your referee is of the opinion from his own experience with this method that the results obtained by it are slightly below the truth, and that one must be very careful not to make the copper solution too acid with acetic acid, or extremely varying results will be obtained.

As to Method II for total copper oxid, here again most of the averages agree well with each other, but are not as close together as one would desire. In most cases individual chemists seem to obtain slightly higher results by this method than by Method I.

LONDON PURPLE.

TABLE III.—*Results of work done with London purple.*

Analyst.	H_2O.	Total As_2O_3.	Total As_2O_5.	Soluble As_2O_3.	Soluble As_2O_5.
	Per ct.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
L. H. Smith, Illinois	4.10	16.85	25.35	10.83	4.58
	4.18	16.63	27.90	....	....
	....	16.56	26.19	10.60	4.73
	....	16.41	27.06	....	....
H. T. Beans, Idaho	4.06	16.60	28.33	10.42	4.08
	4.07	16.54	27.82	10.51	3.54
	....	15.58	....	....	....
T. M. Price, Maryland	3.80	16.50	27.18	10.75	4.25
R. W. Thatcher, Washington	3.64	16.56	27.16	10.63	4.01
	3.63	16.66	27.20	10.68	4.21
D. L. Cleaves, Massachusetts (Hatch)	4.55	16.65	....	....	....
	4.34	16.69	....	....	....
R. J. Davidson, Virginia	4.07	16.84	25.71	10.83	5.36
	4.08	16.79	25.20	10.79	5.42
	....	16.72	25.52	10.57	5.84
	....	16.79	25.20	10.62	....
H. J. Warner, Bureau of Chemistry, U. S. Department of Agriculture.	3.90	16.87	27.36	11.15	3.94
	3.74	16.87	27.19	11.15	3.80
James Emery, Biochemic Division, Bureau of Animal Industry, U. S. Department of Agriculture.	....	16.65	27.45	....	....
	....	16.72	....	....	....
J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture.	3.90	16.64	27.02	11.02	4.00
	3.90	16.78	27.10	11.04	4.20

COMMENTS OF ANALYSTS.

L. H. Smith.—The method for determining total arsenic acid was not satisfactory owing to the dark-colored solution.

R. W. Thatcher.—After repeated trials I was finally able to obtain fairly concordant results in determinations of total arsenic acid. I regard the method as very unsatisfactory, as it is only with great difficulty that the addition of too much thiosulphate is avoided.

In the determination of soluble arsenious and arsenic acids I tried the effect of suspending 1 gram of substance in 1,000 cc of water, with the following results: Soluble As_2O_3=12.19, 12.36 and soluble As_2O_5=6.81, 6.66.

D. L. Cleaves.—It was found impossible to determine the total arsenic oxid, as a satisfactory end point could not be obtained.

DISCUSSION OF RESULTS.

The results obtained by different chemists upon the method for determining total arsenious oxid in London purple leaves little to be desired, and at least show that close results can be obtained by different analysts who employ this method.

The results obtained by using the method for total arsenic oxid, while they vary a good deal, show that this method is worthy of further study. Five analysts obtained results that agree very closely with each other; one obtained results slightly below the general average; one obtained results slightly above the general average; one obtained figures which, separately, were unsatisfactory, but whose average agreed with the figures of the other five, and one was not able to obtain satisfactory results at all. Your referee, who first applied this method to the analysis of London purple, is aware of the fact that the end points are very hard to read, especially for one who has had no experience with the method. He is, however, of the opinion that as one gets more familiar with this method its difficult features disappear, and that one can finally arrive at such a point that identical results, or very closely agreeing results, will be obtained every time.

The results obtained by the method for determining soluble arsenious oxid in London purple agree fairly well. The results obtained by the method for soluble arsenic oxid vary to quite an extent, but with one exception are not widely different from one another.

COPPER CARBONATE.

TABLE IV.—*Results of work done on copper carbonate.*

Analyst.	Copper oxid.		
	Method I.	Method II.	Method by electrolysis.
	Per cent.	*Per cent.*	*Per cent.*
R. J. Davidson, Virginia	58.47	58.23	....
	58.46	58.53	....
	....	58.29	....
H. J. Warner, Bureau of Chemistry, U. S. Department of Agriculture.	58.42	58.53	....
	....	58.96	....
James Emery, Biochemic Division, Bureau of Animal Industry, U. S. Department of Agriculture	58.54	58.96	58.47
	58.54	58.96	58.60
	58.14	58.96	....
J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture	58.24	59.01	....
	58.24	59.01	....

The results obtained by Method I agree with one another much better than they did when this method was applied to Paris green, and seem to leave very little to be desired. Mr. Emery, the associate referee, made electrolytic determinations of the copper in this compound which showed that Method I for such compounds as these gives extremely accurate results. Method II gives results which in three out of the four cases are slightly too high.

POTASSIUM CYANID.

TABLE V.—*Results of work done on potassium cyanid.*

Analyst.	Potassium cyanid.
	Per cent.
R. J. Davidson, Virginia	52.42
	52.42
	52.42
H. J. Warner, Bureau of Chemistry, U. S. Department of Agriculture	52.75
	52.75
James Emery, Biochemic Division, Bureau of Animal Industry, U. S. Department of Agriculture	52.31
	52.31
J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture	52.87
	52.87
	53.12

The results obtained by using the method as outlined for the determination of cyanogen and then calculating the results to potassium cyanid are very good and agree exceedingly well with one another, especially when we take into consideration the fact that it was very difficult to obtain even samples in the beginning, on account of the hygroscopic nature of the substance employed.

SODA LYE.

TABLE VI.—*Results of work done on soda lye.*

Analyst.	Sodium hydrate.		Sodium carbonate.	
	Method I.	Method II.[a]	Method I.	Method II.[a]
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
R. J. Davidson, Virginia	73.12	72.22	3.61	5.28
	73.12	....	3.61	....
H. J. Warner, Bureau of Chemistry, U. S. Department of Agriculture	72.16	70.58	2.29	3.35
	72.16	70.44	2.29	3.35
James Emery, Biochemic Division, Bureau of Animal Industry, U. S. Department of Agriculture	71.60	71.51	4.85	4.36
	71.60	....	4.63	....
	71.62	....	4.63	....
J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture	71.84	70.96	2.37	3.17
	71.84	70.80	2.13	3.17
	....	....	....	3.17

[a] Third normal potassium acid sulphate was used for the titration.

It was exceedingly difficult to prepare the various samples of soda lye so that they would be strictly comparable with one another. Large pieces of the lye were broken up, and these smaller pieces were placed in bottles whose stoppers were covered with paraffin so that the contents would not absorb moisture. Working, however, with as great rapidity as possible, it was not possible to avoid having some samples absorb slightly more moisture and carbon dioxid than others. Taking, then, these facts into consideration, quite good results were obtained upon these samples for sodium hydroxid by both Methods I and II. The results obtained for sodium carbonate are not so good, but very likely this is largely due to the fact that the samples absorbed different amounts of carbon dioxid, and that the analysts failed to use water that was entirely carbon-dioxid-free.

Upon considering each analyst's work, it will be seen that by Method I higher results are obtained for sodium hydroxid than by Method II, while by Method I lower results are obtained for sodium carbonate than by Method II, with the exception of Mr. Emery's figures.

It is the opinion of the referee that the results obtained by Method II are more accurate than the results obtained by Method I, for the following reasons: In Method I, after the carbonates are precipitated out by barium chlorid, the supernatant liquid is titrated for the sodium hydroxid present. Since barium carbonate is soluble to quite an extent in water, that portion which is soluble is also titrated as sodium hydroxid, thus increasing the sodium hydroxid figure. In Method II all of the sodium hydroxid and one-half the sodium carbonate is first titrated, then the other half of the sodium carbonate, so there does not seem to be the same chance for error.

WHALE-OIL SOAP.

TABLE VII.—*Results of work done on whale-oil soap.*

Analyst.	Moisture.
	Per cent.
James Emery, Biochemic Division, Bureau of Animal Industry, U. S. Department of Agriculture	37.41
J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture	37.47
	37.37

The complete work upon soap samples was not undertaken by any of the chemists engaged in the work, but two of them made determinations of the moisture. As will be seen, their results agree very well with each other.

FORMALIN.

TABLE VIII.—*Results of work done on formalin.*

Analyst.	Formaldehyde.				
	Method I.	Method II, by weighing.	Method II, by distillation.	Method III.	Iodometric method.[a]
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
R. W. Thatcher, Washington	36.56		36.91	35.31	36.91
	36.58		37.06	34.91	36.58
	36.76			35.03	36.69
E. F. Ladd, North Dakota				35.53	
				35.53	
				35.45	
James Emery, Biochemic Division, Bureau of Animal Industry, U. S. Department of Agriculture.	37.95				
	38.00				
J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture.	37.18	36.50		36.20	
	37.12	36.16		36.85	

[a] Taken from Allen's Com. Org. Analysis, vol. 1, p. 229.

COMMENTS BY ANALYSTS.

R. W. Thatcher.—In Method II, I attempted to dry and weigh the hexa-methyl-tetramine formed by evaporating at 80° C. until the mixture began to sputter and then placing in a vacuum desiccator over sulphuric acid, but found that a month's drying in this manner had not removed half the remaining moisture, hence I abandoned this method as impracticable. I also made three determinations on this sample by the iodometric method.

E. F. Ladd.—Method I gave very unsatisfactory results. We tried the best grades of hydrogen-peroxid that could be found in the market, but could not secure concordant results. Ours ranged from 22 per cent to about 33 per cent. Method II proved wholly unsatisfactory in our hands, as both of these methods have with us in the past.

J. K. Haywood.—In Method II the dishes containing the hexa-methyl-tetramine continued to lose weight for three weeks, and were still losing weight when the determination was stopped. At the end of almost two weeks the loss each day was practically constant, and at this time the weight of the residue in the dishes showed the presence of 36.66 and 36.45 per cent formaldehyde. After about three more days the weight had decreased to such an extent as to make the figures for formaldehyde 36.50 and 36.16 per cent. It was very hard to read the end point in Method III.

SUGGESTIONS.

From the above work done upon formalin and the comments made by the various chemists engaged in the work, your referee is of the opinion that Method II (by weighing the residue) is nearly valueless and should be abandoned. It also hardly appears best to do further work upon Method III. It appears, then, that the two most promising methods are I and II (by distilling off the excess of ammonia). It might also be well to test the iodometric method referred to by Mr. Thatcher, and the cyanid method referred to by Mr. Houston last year.

TOBACCO EXTRACT.

TABLE IX.—*Results of work done on tobacco extract.*

Analyst.	Nicotine.					
	Method I.		Method II.		Method III.	
		Per cent.		*Per cent.*		*Per cent.*
R. J. Davidson, Va	3 distillations	26.20	After 24 hours standing	34.88	Ran as directed	14.71
	4 distillations	26.40	After 72 hours standing at 40° C.	26.61	Extra extraction	15.18
	4 distillations	26.40	After 72 hours standing at 40° C.	28.40	Extra extraction	21.00
	5 distillations	26.49	After 48 hours standing and heating to 40° C. for 72 hours	26.40	Extra extraction	16.65
					Extra extraction	23.67
James Emery, Biochemic Division, Bureau of Animal Industry, U. S. Department of Agriculture.	Repeated distillation and small amount of substance taken	27.83	After 36 hours standing in breakers	25.65	Ran as directed	17.64
				25.75		18.14
				26.87	Extra extraction	21.41
						21.30
J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture.		28.04	After 36 hours standing	28.45	Ran as directed	19.92
		25.73				18.24
		25.79			Extra extraction	23.42
		26.52				24.18

COMMENTS BY ANALYSTS.

R. J. Davidson.—By running Method II exactly as directed, ammonia was still present, so large results were obtained. By allowing the vessel to stand seventy-two hours at 40° C., ammonia was absent and good results were obtained. A duplicate of this gave high results. One that stood forty-eight hours and then seventy-two hours at 40° C. gave good results.

By Method III, when this was run exactly as described, very low results were obtained; when it was extracted with 12, 10, 10, 10 and 10 cc, 15.18 per cent was obtained; when it was extracted with 12, 11, 10, and 98 cc, 16.65 per cent was obtained, and when it was extracted with 12, 11, 10, and 98 cc, and then with 12, 11, and 10 cc, 23.67 per cent was obtained.

James Emery.—Instead of following directions exactly for Method I, a smaller amount of the extract was weighed out than is directed in the method. The distillation was continued until only a small amount remained in the distillation flask, then the distillating flask was refilled with water and again distilled with steam. For Method II, the extract was weighed out in beakers instead of flasks to allow the ammonia to escape and allowed to stand with the concentrated sodium hydroxid for thirty-six hours. When Method III was run as directed, low results were obtained, but where the magma was extracted three additional times, with 50 cc each time, results of 21.41 per cent and 21.30 per cent were obtained. No more could be extracted.

J. K. Haywood.—After standing with sodium hydroxid for thirty-six hours, Method II gave the result submitted, but a very slight smell of ammonia seemed to be present. When Method III was run as directed, very low results were obtained. The magma was extracted twelve extra times with 10-cc portions of petroleum-ether, and the results 23.42 per cent and 24.8 per cent as given were obtained.

The sample of extract sent out this year was extremely difficult to work upon, since it contained a very great excess of ammonia and ammonium salts. It is not often that one would be called upon to analyze a sample that presented the difficulties which this one did.

Your referee is of the opinion that if Method I is properly carried out, fair results may be obtained, and thinks that good results may also be obtained by Method II if one adopts the scheme devised by Mr. Emery, of weighing the extract out in beakers and allowing it to stand in these in contact with concentrated sodium hydroxid for about forty-eight hours. Method III evidently gives low results, as has been shown in these experiments and in work done by one of our former referees.

In conclusion, I desire to express my thanks to those who have so kindly cooperated with me in this work. Especially my thanks are due to Mr. Emery, the associate referee.

Mr. F. W. Traphagen, of the Montana Experiment Station, sent in figures upon various of these groups but unfortunately they were received too late to be incorporated in the report.

RECOMMENDATIONS.

I would respectfully recommend:

(1) That, as Method I, for total arsenious oxid in Paris green has been thoroughly tested and shown to give good results, and as your referee has thoroughly tested this method, compared it with other methods, and always found that it gave most excellent results, it be adopted as one of the official methods of the A. O. A. C.

(2) That Method II for total arsenious oxid in Paris green be tested next year upon samples of Paris green that contain a large amount of free arsenious oxid.

(3) That both methods for soluble arsenious oxid in Paris green be further tested,

and that the referee try to so modify the directions that more concordant results may be obtained.

(4) That Methods I and II for copper oxid in Paris green be further tested and compared with an electrolytic determination of the copper oxid.

(5) That all methods of analysis of London purple be further tested.

(6) That Methods I and II for determining copper oxid in copper carbonate be compared with the electrolytic method.

(7) That the cyanid method be tested upon samples of known strength or compared with some well-known gravimetric method.

(8) That both the methods of analysis of soda lyes be further tested, using lyes which contain a larger percentage of sodium carbonate.

(9) That the methods of analysis of soaps be further tested.

(10) That Method II for formalin (by weighing the residue) and Method III be dropped and that Methods I and II (by distillation) be further tested and compared with the cyanid and iodometric methods.

(11) That Methods I and II for nicotin be further tested upon extracts containing both large and small amounts of nicotin, and that Method III be dropped for determining large amounts of nicotin, but tested upon extracts containing small amounts of nicotin.

The **President**: Discussion is now in order.

Mr. **Davidson**. In regard to the iodin solution, I would like to ask the referee if it would be possible to make it half strength. It makes a considerable difference in the percentage by weakening the solution just a little.

Mr. **Haywood**. In connection with that I will say that I think it might be a very good idea to make the iodin solution weaker than it is. I have noticed, myself, that 0.2 cc in the burette reading makes a great difference in the percentage, and anyone is apt to make an error of 0.1 to 0.2 cc in this reading.

Mr. **Davidson**. In regard to the nicotin determinations in tobacco extracts, I would state that this sample was very rich in ammonia, and I, like Mr. Emery, used a beaker in which to treat the extract with sodium hydrate instead of a flask. I think this is absolutely necessary because of the large amount of ammonia present.

Mr. **Haywood**. It is undoubtedly shown by all of our work that the expulsions of ammonia by sodium hydrate must be effected in an open vessel. I believe that when Mr. Winton originally got out this method he worked upon extracts that did not contain such large amounts of the ammonia and ammonium salts as the sample sent out by us, and therefore his method will have to be modified to some extent to give good results with such extracts as the one worked upon. As Mr. Winton, the originator of this method, is here, I would like to ask him if he worked upon samples containing large amounts of free ammonia.

Mr. **Winton**. I do not remember about the ammonia, but I do remember that the process did not seem to give as sharp an end point as the old Kissling method. I devoted my attention more to that point than to the study of the effect of varying quantities of ammonia upon the determination.

Mr. **Haywood**. In connection with this study on insecticides, I desire to call attention to some work that we are now carrying on in collaboration with the Division of Entomology on the effect of varying amounts of soluble arsenic upon the foliage of different kinds of trees. We found in working with Paris green that there are three conditions that may give rise to the scorching of the foliage by the soluble arsenic present in the green: First, the arsenious oxid which exists as such in the Paris green is injurious and scorches the foliage; second, there are certain greens which are badly made and which in consequence are soon broken up by the moisture and carbon dioxid in the air, thus releasing free arsenious oxid; third, if the green is very fine it is more easily broken up by water, and consequently free arsenious oxid is set free, which injures the foliage. Our preliminary work upon this subject indicates that apple and pear trees can stand Paris green containing from 7 to 8 per cent of free arsenious oxid, while peach trees can stand Paris green containing about 4 per cent. I will say also in this connection that all the determinations of arsenious oxid were made by the ten-day extraction method and are consequently much higher than the results would have been by using a twenty-four-hour extraction, as was done by many chemists in past years.

Mr. **Davidson**. I do not know whether it is necessary to make a motion, but if it is I would like to make one as follows: That the referee for next year be instructed to try the effect of changing the strength of the iodin solution in the determination of total arsenic in Paris green.

Mr. **Haywood**. I second the motion.

The motion was adopted.

Mr. **Davidson**. In connection with the nicotin determination, I would also like to make a motion that this point we have just spoken of, concerning the desirability of allowing the extract to stand in beakers instead of flasks, be called to the attention of the referee for next year.

Mr. **Haywood**. I second the motion.

The motion prevailed.

Mr. **Winton**. With reference to the question of large amounts of ammonia in nicotin extracts, I will say that I have had success in removing this ammonia by placing the dish containing the extract under a bell jar with a dish containing strong sulphuric acid and drawing a current of air over the dishes.

Mr. **Haywood**. I have tried the method suggested by Mr. Winton and have found that it works quite satisfactorily. I think the Winton method as well as the Lloyd method will have to be modified to quite an extent in order that they give good results upon substances containing such a large amount of nicotin. They were both evidently originally devised for extracts which contain only small amounts of nicotin and little ammonia. It would very likely be best not to make any cast-iron rules for the chemists to follow. In the Winton method, for example, it might be directed that the extract be allowed to stand in contact with sodium hydrate until all ammonia is removed, and in the Lloyd method that the extractions with ether be continued until all nicotin is extracted from the magma.

Mr. WILEY. There is a point that I would like to make right here in connection with this discussion. It seems to me that the time has passed when we must give minute details and directions to analysts as to how they are to carry on the work. It would be much better to give a broad outline of the plan and let each analyst work out the details for himself. When this association first began its work minute details as to manipulation were undoubtedly necessary, but our men have now been educated up to the point where they are expert analysts and will no doubt obtain better results by being allowed to work out the details for themselves. In other words, it is not the work of the analyst that we wish to test, but the working of the method.

The PRESIDENT. If there is no objection, the report of the chairman of the committee on fertilizer legislation will be called for at this time.

Mr. WILEY. The only report that I have to make on this subject is the one that the president of the association has prepared.

REPORT OF COMMITTEE ON FERTILIZER LEGISLATION.

By H. J. WHEELER.

As chairman of the committee on uniform fertilizer legislation of the Association of American Agricultural Colleges and Experiment Stations I will say that the matter of fertilizer legislation was brought up at the instance of our committee before the United States Commission on Uniform Legislation at its last meeting and the members from the respective States were instructed by a resolution to that effect to assist in so far as they could in securing uniformity of legislation along the lines laid down by this association and the Association of American Agricultural Colleges and Experiment Stations. As chairman of the committee for the latter association I have been consulted by several States in regard to fertilizer laws. Something is being done continually to bring about uniformity in legislation in regard to fertilizers.

Mr. WILEY. I move that the report be accepted and the committee continued.

The motion was adopted.

The PRESIDENT. Are there any business matters to come before us at this time?

Mr. WILEY. I would like to make one or two announcements. The agent of the Trunk Line is here to validate tickets. The tickets of those who came over the southeastern line he has taken down to the Southern Railway office.

I will state that the chiefs of the laboratories of the Bureau of Chemistry are present and will be glad to receive members of this association and to show them through our laboratories after the adjournment of this meeting.

The **President**. The association will bear in mind that in regard to the recommendations of referees matters of minor importance and matters that are urgent may be acted upon at this time by a two-thirds vote. I will ask the chairman of Committee A on recommendations if there are any such matters to be considered.

Mr. **Street**. I think that the majority of the recommendations made to us fall within this scope. We are ready to report on all of our sections except insecticides.

REPORT OF COMMITTEE A ON RECOMMENDATIONS.

SOILS.

Mr. **Street**. It is recommended—

That the N/200 hydrochloric acid method be given further trial.

That samples be so taken that it will be possible to determine the available potash and phosphoric acid in each stratum sampled to a total depth of 3 or 4 feet.

That the methods for the analysis of alkali soils be referred to the referee, with instructions to determine the most desirable amount of soil and the amount of water to be used in dissolving the soluble salts.

That in the selection of soils for examination, virgin soils be given a place among the soils worked upon.

That the quantitative method for the determination of the acidity of soils be made a provisional method.[a]

Mr. **Van Slyke**. I move that the recommendations be adopted as read.

Mr. **Hopkins**. I notice that the recommendations include an arbitrary division as to the depth of the different strata to be taken in determining available phosphoric acid. The provisional method for soil sampling which was adopted last year states that we shall sample distinctly different strata, rather than make an arbitrary division. You are all aware that we frequently find soils that extend to the depth of 11 to 17 inches. The character of the soil changes very much, and it seems to me that it might be well to modify the provisional method for soil sampling, if the recommendation could be modified to include the provisional method.

Mr. **Street**. I think that is the idea of the recommendation. We would be willing to incorporate that.

Mr. **Hopkins**. I make a motion to so amend the provisional method relating to the sampling of soils. I would like to ask also that the method for determining the acidity of soils be adopted as a provisional method, and that it be referred to the referee for investigation next year. A number of members have spoken to me regarding the details of the method. I am sure a considerable amount of work will be done in this line, and, as we have no method in the association for such determinations, I think perhaps it would be well if this could be adopted as a provisional method and further investigation encouraged next year. I will move to further amend the report to that effect.

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[a] As amended.

The motion prevailed, and the original motion, as amended, was then adopted.

POTASH.

Mr. **Street**. It is recommended—

That further experiments be conducted for the purpose of securing a method that will dissolve all of the potash in mixed fertilizers, giving attention to the amount of material used and the amount of water used for making the solution.

That the referee, if he have sufficient time, study methods for the determination of moisture in potash salts.

Mr. **Van Slyke**. I move that the recommendations, as read, be adopted.

This was carried.

ASH.

Mr. **Street**. It is recommended—

That the referee be requested to make a further study of the fusion method for the determination of sulphur, and also methods to determine how much of the sulphur belongs to the ash.[a]

That the method of determining chlorin by ignition with sodium carbonate be adopted as a provisional method.

Mr. **Wiley**. It seems to me that there is a very important point under ash which the committee ought to consider. I do not know that I am prepared to amend the report in any way, but the question of determining the sulphur in the ash of a plant ought to be considered. All these fusion methods have for their object the determination of total sulphur in the form of sulphuric acid. I know that almost all the sulphur that exists in the plant is not properly found in the ash, but I think the referee on ash ought to work on a method to determine how much of the sulphur does belong to the ash. So I would like to suggest, and if it meets with the approval of the association it would be advisable to make a motion, that the referee be instructed to investigate methods by which we can determine how much of the sulphur in plants and plant products belongs to the ash. Of course the remainder belongs to the organic compound. I move that the report of the committee be amended to this extent, that the referee be instructed to investigate the method of determining what part of the sulphur in plants and plant products should be considered in the ash.

The amendment was carried, and the report on ash was adopted as amended.

NITROGEN.

Mr. **Street**. Under nitrogen there are two recommendations—

That the subject of the availability of organic nitrogen be further studied with the view to so perfecting the neutral and alkaline permanganate methods as to secure concordant results.

That it be earnestly urged that, in reporting the analyses of complete fertilizers, the amounts of nitrogen existing in the form of nitrates, ammonia salts, and organic nitrogen be specifically stated.

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[a] As amended.

On motion, the recommendations on nitrogen were adopted.

Mr. STREET. Our only report on phosphoric acid is that the committee desires to get the sense of the association as to the advisability of discontinuing the investigations on basic slag. From the representation of the committee, made from our experience, it seems that the use of basic slag is extremely limited. It was suggested that the association should not work on the matter, as the slag is so little used.

The PRESIDENT. Does the association desire to take any action in this matter?

Mr. HOPKINS. I would say that a firm in Philadelphia is buying considerable basic slag which it is putting out. It seems that the use of this material is on the increase.

GENERAL RECOMMENDATION.

Mr. STREET. We have one general recommendation, which was considered by the referees, which reads as follows:

In the comparison of methods, in addition to the general letter requesting cooperation, it is recommended that the referee invite the assistance of workers experienced in the special portions of the work assigned to him.

This recommendation was adopted.

[NOTE BY THE EDITOR.—The following recommendations were approved by Committee A, after the adjournment of the meeting, and forwarded by the chairman with the following comment:]

DECEMBER 13, 1902.

DEAR SIR: In reply to your letter of December 11, I inclose herewith the balance of the report of Committee A on recommendations, covering the recommendations of the referee on insecticides. You will notice that the first recommendation alone entails any change in methods, and the committee in this case has accepted the long experience of Mr. Haywood in this kind of work as strong evidence that the change is a worthy one. The other recommendations refer merely to details or suggestions to the new referee.

Very respectfully, JOHN PHILLIPS STREET, *Chairman.*

INSECTICIDES.

(1) That, as Method I, for total arsenious oxid in Paris green has been thoroughly tested and shown to give good results, and, as your referee has thoroughly tested this method, compared it with other methods, and has always found that it gave most excellent results, it be adopted as one of the official methods of the A. O. A. C.

(2) That Method II for total arsenious oxid in Paris green be tested next year upon samples of Paris green that contain a large amount of free arsenious oxid.

(3) That both methods for soluble arsenious oxid in Paris green be further tested and that the referee try to so modify the directions that more concordant results may be obtained.

(4) That Methods I and II for copper oxid in Paris green be further tested and compared with an electrolytic determination of the copper oxid.

(5) That all of the methods of analysis of London purple be further tested.

(6) That Methods I and II for determining copper oxid in copper carbonate be compared with the electrolytic method.

(7) That the cyanide method be tested upon samples of known strength or compared with some well-known gravimetric method.

(8) That both the methods of analysis of soda lyes be further tested, using lyes which contain a larger percentage of sodium carbonate.

(9) That the methods of analysis of soaps be further tested.

(10) That Method II for formalin (by weighing the residue) and Method III be dropped, and that Methods I and II (by distillation) be further tested and compared with the cyanid and iodimetric methods.

(11) That Methods I and II for nicotin be further tested upon extracts containing both large and small amounts of nicotin and that Method III be dropped for determining large amounts of nicotin, but tested upon extracts containing small amounts of nicotin.

JOHN PHILLIPS STREET.
M. E. JAFFA.
B. B. ROSS.
E. G. RUNYAN.
F. P. VEITCH.

The **PRESIDENT**. I will call on the chairman of Committee B on recommendations for his report.

REPORT OF COMMITTEE B ON RECOMMENDATIONS.

Mr. **KRUG**. With reference to dairy products, there are no changes in the method. The committee would, however, recommend that the referee for next year make a study of the effects of preservatives on the albumin of milk.

Mr. **WILEY**. I would like to ask a question. Does that mean the albumin proper or all of the protein of the milk. I infer that it means all of the protein.

Mr. **KRUG**. If the referee on dairy products were here, he could inform you. I am not familiar with the subject.

The recommendation was adopted as read.

FEEDING STUFFS.

Mr. **KRUG**. With reference to feeding stuffs the committee recommends that from the sixteenth line,[a] the method be changed to read as follows, following the directions prescribed by Kröber:

The solution is made up to 400 cc with 12 per cent hydrochloric acid, and allowed to stand overnight. The amorphous black precipitate is filtered into a tared gooch through an asbestos felt, washed carefully with 150 cc of water in such a way that the water is not entirely removed from the crucible until the very last, then dried to constant weight by heating four hours at 100° C., cooled, and weighed in a weighing bottle, the increase in weight being reckoned as phloroglucid. To calculate the furfurol from the phloroglucid, use the following formulæ:

For weight of phloroglucid (a) under 0.03 grams.

$$\text{Furfurol} = (a + 0.0052) \times 0.5170.$$
$$\text{Pentoses} = (a + 0.0052) \times 1.0170.$$
$$\text{Pentosans} = (a + 0.0052) \times 0.8949.$$

For weights of phloroglucid (a) from 0.03 grams to 0.300 grams Kröber's table is recommended.[b] If this is not available, use the formulæ:

$$\text{Furfurol} = (a + 0.0052) \times 0.5185.$$
$$\text{Pentoses} = (a + 0.0052) \times 1.0075.$$
$$\text{Pentosans} = (a + 0.0052) \times 0.8866.$$

For weights of phloroglucid (a) over 0.300 grams.

$$\text{Furfurol} = (a + 0.0052) \times 0.5180.$$
$$\text{Pentoses} = (a + 0.0052) \times 1.0026.$$
$$\text{Pentosans} = (a + 0.0052) \times 0.8824.$$

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[a] Circular 7, Div. of Chem., U. S. Dept. Agr. Provisional method for the determinations of pentosans by means of phloroglucol.

[b] Journal für Landwirtschaft 48: 1900, p. 379.

Mr. **Krug**. I think it would be advisable to make these changes at this time.

Mr. **Wiley**. Does this involve a change in the method?

The **President**. The Chair understands that this is a change of vital importance at this time. Is there anything to be said upon the adoption of this?

Mr. **Browne**. Is this a provisional method or an official method?

Mr. **Krug**. This is a provisional method.

On motion the recommendation was adopted.

Mr. **Krug** (continuing report). The next recommendation in connection with this same method is the adoption of Kröber's factors.

Mr. **Wiley**. How much do they differ from the old factors?

Mr. **Browne**. Very little. I think we ought to adopt these factors.

On motion of Mr. Krug, the recommendation was adopted.

TANNIN.

Mr. **Krug** (continuing report). I understand that we have here changes in the official method on tannin[a] that must go over until next year. I would like to read them to the association if there is no objection.

To omit the moisture determinations in extracts.

To change (*b*) in paragraph III so as to read:

Evaporations shall take place under precisely the same conditions as to contact with steam or with a metallic plate; all dryings called for after evaporation shall be done by one of the following methods under precisely the same conditions, so that the different residues of each analysis may occupy the same shelves in the drying oven.

1. For eight hours at the temperature of boiling water in steam oven.
2. For six hours at 100° C. in air bath.
3. For five hours at 100° C. in vacuo.

V. Soluble solids to read as follows:

Single pleated filter paper (S and S No. 590, 15 cm) shall be used. To 2 grams kaolin add 75 cc of the tanning solution, stir, let stand fifteen minutes and decant as much as possible (not on the filter), add 75 cc of the solution, stir, and pour on the filter. Keep filter full, reject the first 150 cc of filtrate, evaporate, and dry next 100 cc. The portion dried for determination shall be perfectly clear and evaporation during filtration must be guarded against.

VI. Non-tannins: Two grams kaolin to be used when filtering.

Omit paragraph VIII on testing hide powder and section (*b*) of paragraph IX, on testing nontannin filtrate for soluble hide.

The following provisional method for the determination of total acidity in liquors is recommended:

Place 100 cc of the liquor in a 500-cc flask and make up to the mark with water. To 100 cc of diluted liquor in a flask with tube condenser add 2 grams of chemically pure animal charcoal. Heat to boiling temperature with frequent shaking, cool, filter, and titrate an aliquot portion with decinormal alkali.

The following suggestions are made by the referee on tannin with reference to next year's work:

Additional experimental work on soluble solids, acid determinations, and on the amount of extract taken for analysis.

A comparative study with the following modifications of the charcoal method is recommended: To 100 cc of diluted liquor, prepared as in the provisional method, add 2.5 grams chemically pure animal charcoal, stir every fifteen minutes for two hours, filter, and titrate an aliquot portion with decinormal alkali.

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[a] Subsequent investigation showed that this method had never been adopted as official, and therefore these changes in a provisional method became effective for the work of 1902–1903.

The referee also recommends that the work during the next year shall include a study of the correction method for the tannin absorption of the filter paper which was adopted at the last meeting of the International Association of Leather Trades Chemists, held at Leeds, England.

The **President**. Is it the desire of the association that action be taken?

Mr. **Krug**. I think it would be well to have these recommendations held over.[a]

If permissible at this time, I would like to say that the report of the European Leather Trades Chemists Association, to which I referred yesterday, was received this morning, and I make a motion that the secretary be authorized to incorporate an extract of the report in the proceedings of this association.

[Note by the Editor.—The following extract from this report is inserted in accordance with the above instructions:]

At the last convention of the International Association of Leather Trades Chemists the following resolutions, which embody the changes made in the international method, were adopted:

(A) The filtration of the solution for analysis may take place through any paper which may be considered more suitable for the particular case, and with or without the use of kaolin, the absorption of the tanning matter being corrected for by an amount determined by a similar filtration of a clear solution. Perfectly clear solutions need not be filtered.

Following the discussion on the filter paper in tanning analysis the following resolution was carried:

(B) To determine the correction, about 500 cc of the tanning solution of the strength prescribed for analysis is obtained perfectly clear, preferably by the method of filtration, which is to be corrected for. After thorough mixing, 50 cc is evaporated to determine "total soluble No. 1." A portion of the remainder is then filtered in the manner for which correction is to be made, and 50 cc of the filtrate is evaporated for "total soluble No. 2." Deducting No. 2 from No. 1 the difference is the correction required, which must be added to the "total soluble" found by analysis. It is generally advisable, both in analysis and in the second filtration for correction, to filter first 150 cc, which in analysis may be used for determination of nontannins, and then to employ the next 50 cc for evaporation, keeping the filter full during the operation; but whatever procedure is adopted must be rigidly adhered to in all analyses to which the correction is applied. Where kaolin is employed a constant weighted quantity (1 or 2 grams) must be used, which is first washed with 75 cc of the liquor by decantation, and then washed onto the filter with a further quantity of liquor, of which 200 cc is filtered as above.

(C) In making samples of spent tans in cases where the prescribed strength can not otherwise be obtained, it is permissible to concentrate the whole solution. It is advisable, where ready means exist, to concentrate in vacuo, failing the necessary apparatus, concentration may be carried out by evaporation in a flask, in the neck of which a funnel is placed to prevent oxidation.

(D) That the filter method of determination of nontannins, as prescribed in the rules of the association, remain the official method until the next conference, but that members be permitted to employ the chrome hide-powder method of the conference of the American Association of Official Agricultural Chemists in 1901 where it is desired, the fact being clearly stated on the report that the A. O. A. C. method has been employed, and not that of the I. A. L. T. C.

(E) Used liquors must be analyzed by the A. O. A. C. method of 1901.

The **President**. If there is no further report from Committee B, the Chair will call for the report of Committee C on recommendations.

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[a] As a circular letter addressed to the executive committee procured permission to insert the word "provisional" over methods on tannin in Bulletin 46, the changes above noted have been considered by the referee as a part of the provisional methods.

REPORT OF COMMITTEE C ON RECOMMENDATIONS.

FOOD ADULTERATION.

Mr. **Leach**. The committee has only one recommendation to make, namely, that the association empower the referee on food adulteration to assign the work as he may see fit, acting on the advice of the executive committee. In this work the number of subjects is greatly in excess of the number of men who can work upon them, and it is desired to further subdiyide the work of the coming year. It is not possible at present to definitely state what the work will be and how it will be divided. The recommendation of the committee is that the association authorize the referee to appoint such associate referees as he may see fit, to take part in this work and make such subdivisions of the subject as may seem best. The committee further earnestly recommends that in view of the scarcity of men to do the work, a special invitation be sent out by the executive committee to induce all food chemists to work along these lines and to joint this association.

On motion of Mr. Leech, the recommendation was adopted.

Mr. **Munson**. I would like to ask what action has been taken on the recommendations of the referee on sugar.

The **President**. These recommendations have been overlooked. If there is no objection they will be assigned to Committee B, and a report will be written so that action can be taken next year.[a]

Mr. **Street**. I am afraid that our committee has made an error in acting on the nitrogen recommendation. Should they have been referred to some other committee? We shall be glad to be instructed by the convention.

The **President**. It is understood by the association that nitrogen is assigned to Committee A.

Mr. **Van Slyke**. Recommendations on the subject of separation of nitrogenous bodies should be referred to the committee on foods or on dairy products. In the case of the recommendation made on this subject in the paper read yesterday, I move that it be referred to Committee B having charge of diary products.[a]

The motion was adopted.

Mr. **Leach** (continuing report on food adulteration). There is one other point that I would like to speak of, and that is the committee on food adulteration has made some slight changes in the provisional methods as printed in Bulletin No. 65, United States Department of Agriculture, Bureau of Chemistry, and it is recommended that these be adopted as part of the provisional methods. This bulletin was discussed and criticised by the food referees, submitted to the association, and adopted last year as provisional, but subsequent experience has dictated certain changes, and we ask that these changes be incorporated in the provisional methods.

Mr. **Munson**. I move the adoption of this recommendation.

The motion was adopted.

The **President**. I will now call on Mr. Van Slyke for the report of the committee on resolutions.

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[a] As these references were made shortly before adjournment, action was deferred on the recommendations until the meeting of 1903, when the report of chairman of Committee B will be completed.

REPORT OF COMMITTEE ON RESOLUTIONS.

Mr. VAN SLYKE. No resolutions have been reported to the convention, but as chairman I take the responsibility of submitting the usual resolutions which are presented at the close of the convention:

Resolved, That we extend to the Secretary of Agriculture our hearty thanks for the continued assistance which he has so generously given to this association.

Resolved, That we acknowledge the courtesy of the authorities of the Columbian University, and thank them for giving us the use of this room for holding the sessions of our convention.

Resolved, That a committee of three be appointed as referees to take charge of the work relating to separation of nitrogenous compounds, the work to be distributed among the members of the committee on the following lines of division: (1) Meat proteids; (2) vegetable proteids; (3) milk and cheese proteids.

These resolutions were adopted.

Mr. WILEY. I move that we offer a resolution of thanks to the president of this association for the impartial manner in which he has conducted this convention.

The motion prevailed.

At 11.30 a. m. the convention adjourned sine die.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1903.

PRESIDENT.

Mr. R. J. DAVIDSON, Blacksburg, Va.

VICE-PRESIDENT.

Mr. M. E. JAFFA, Berkeley, Cal.

SECRETARY.

Mr. H. W. WILEY, Washington, D. C.

ADDITIONAL MEMBERS OF EXECUTIVE COMMITTEE.

Mr. C. L. PENNY, Newark, Del.
Mr. A. E. LEACH, Boston, Mass.

REFEREES.

Phosphoric acid: B. H. Hite, Morgantown, W. Va.
Nitrogen:
- Determination of nitrogen: Fred W. Morse, Durham, N. H.
- Separation of nitrogenous bodies (milk and cheese proteids): L. L. Van Slyke, Geneva, N. Y.

Potash: F. B. Carpenter, Richmond, Va.
Soils: F. P. Veitch, Washington, D. C.
Dairy products: George Cavanaugh, Ithaca, N. Y.
Foods and feeding stuffs: F. D. Fuller, Geneva, N. Y.
Sugar: G. L. Spencer, Washington, D. C.
Tannin: George A. Kerr, Damascus, Va.
Insecticides: J. K. Haywood, Washington, D. C.
Ash: G. S. Fraps, Raleigh, N. C.

ASSOCIATE REFEREES.

Phosphoric acid: C. B. Williams, Raleigh, N. C.
Nitrogen:
- Determination of nitrogen: Edward B. Holland, Amherst, Mass.
- Separation of nitrogenous bodies:
  - Vegetable proteids: R. Harcourt, Guelph, Canada.
  - Meat proteids: W. D. Bigelow, Washington, D. C.

Potash: M. G. Donk, Tallahassee, Fla.
Soils: C. C. Moore, Washington, D. C.
Dairy products: G. E. Patrick, Washington, D. C.
Foods and feeding stuffs: L. H. Smith, Urbana, Ill.

Food adulteration:

(1) Colors: A. S. Mitchell, Milwaukee, Wis.
(2) Saccharine products, including confectionery: Elton Fulmer, Pullman, Wash.
(3) Fruit products: L. S. Munson, Washington, D. C.
(4) Wine: Geo. E. Colby, Berkeley, Cal.
(5) Beer: H. E. Barnard, Concord, N. H.
(6) Distilled liquors: C. A. Crampton, Washington, D. C.
(7) Vinegar: W. M. Allen, Raleigh, N. C.
(8) Flavoring extracts: A. L. Winton, New Haven, Conn.
(9) Spices: R. E. Doolittle, Lansing, Mich.
(10) Baking powder and baking chemicals: F. T. Green, San Francisco, Cal.
(11) Meat and fish: M. E. Jaffa, Berkeley, Cal.
(12) Fats and oils: L. M. Tolman, Washington, D. C.
(13) Dairy products: A. E. Leach, Boston, Mass.
(14) Cereal products: A. McGill, Ottawa, Canada.
(15) Infants' and invalids' foods: H. W. Wiley, Washington, D. C.
(16) Vegetables: G. W. Cavanaugh, Ithaca, N. Y.
(17) Condiments (other than spices): J. O. La Bach, Lexington, Ky.
(18) Cocoa and cocoa products: E. N. Eaton, Chicago, Ill.
(19) Tea and coffee: W. H. Ellis, Toronto, Canada.
(20) Preservatives: W. D. Bigelow, Washington, D. C.

Sugar:

Optical methods: L. M. Tolman, Washington, D. C.
Chemical methods: L. S. Munson, Washington, D. C.
Special analytical methods used in the sugar industry: David L. Davoll, Caro, Mich.

Tannin: H. C. Reed, Stamford, Conn.
Insecticides: James Emery, Washington, D. C.
Ash: F. T. Shutt, Ottawa, Canada.

COMMITTEE ON FOOD STANDARDS.

Mr. Wm. Frear, State College, Pa., chairman.
Mr. H. W. Wiley, Washington, D. C.
Mr. H. A. Weber, Columbus, Ohio.
Mr. M. A. Scovell, Lexington, Ky.
Mr. E. H. Jenkins, New Haven, Conn.

COMMITTEE ON FERTILIZER LEGISLATION.

Mr. H. W. Wiley, Washington, D. C., chairman.
Mr. B. W. Kilgore, Raleigh, N. C.
Mr. H. B. McDonnell, College Park, Md.
Mr. H. A. Huston, Lafayette, Ind.
Mr. B. B. Ross, Auburn, Ala.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statement of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. All persons eligible to membership shall become members ex officiis and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the executive committee, at the regular annual meeting, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 74.

H. W. WILEY, Chief.

THE INFLUENCE OF SOIL AND CLIMATE UPON THE COMPOSITION OF THE SUGAR BEET, 1901.

BY

HARVEY W. WILEY,
CHIEF OF BUREAU.

IN COLLABORATION WITH THE WEATHER BUREAU AND THE AGRICULTURAL
EXPERIMENT STATIONS OF INDIANA, IOWA, KENTUCKY, MICHIGAN,
NEW YORK, UTAH, VIRGINIA, AND WISCONSIN.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., December 31, 1902.

SIR: I have the honor to transmit for your approval manuscript and graphic charts embodying the results of the cooperative work conducted by this Bureau in the study of the effect of environment upon the composition of the sugar beet during the year 1901. To the work as conducted in 1900, the results of which were published in Bulletin 64, Bureau of Chemistry, has been added the consideration of the influence of the soil. I recommend that this manuscript be published as Bulletin No. 74, Bureau of Chemistry.

I wish again to express my appreciation of the work done by the various stations taking part in the experiment, the continued cordial cooperation of the Weather Bureau, which is of vital importance, and the information received from the Coast and Geodetic Survey and the Naval Observatory in response to the request made for geodetic data. The analytical work done in this Bureau was performed, under the direction of Mr. G. L. Spencer, by the assistants in the sugar laboratory, namely, H. W. Houghton, A. W. Bache, and Arthur Given.

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary.

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THE INFLUENCE OF SOIL AND CLIMATE UPON THE COMPOSITION OF THE SUGAR BEET, 1901.

ORGANIZATION OF COLLABORATIVE WORK.

In continuation of the cooperative experimental work with sugar beets carried on in 1900[a] by this Bureau in collaboration with certain experiment stations and the Weather Bureau, the following letters were addressed to the experiment stations of North Carolina, Iowa, Michigan, Utah, Indiana, Wisconsin, Kentucky, and to the two New York stations in the spring of 1901:

March 18, 1901.

Dear Sir: For the collaborative work in the study of the influence of environment on the composition of the sugar beet for the present year, I have decided to use seed No. 5772, Dippe's Kleinwanzlebener Elite, and 20 pounds of this seed marked "special" have been sent to you for such collaborative work.

The area planted need not exceed an eighth of an acre, unless you desire a larger area or a number of plots. This matter is left entirely to your own judgment, and the residue of the seed you can dispose of as you like. I suggest, however, that the special plot be seeded very heavily, so as to be sure of a good stand, and that enough of the seed be reserved for replanting in case the first planting should not germinate.

I will send you in a few days a blank giving some special points in regard to the study of the environment which I should be glad to have you observe during the season.

H. W. Wiley, *Chemist.*

March 18, 1901.

Dear Sir: In connection with the collaborative study of the influence of environment on the composition of the sugar beet, I desire to make a careful chemical and physical analysis of the soils of the plots used for the growing of the beets.

I therefore ask that you take a representative sample of the soil and subsoil of the plot on which you grow the No. 5772 "special" seed during the present year. After getting such a sample, reduce it in size by quartering or otherwise, so as to secure a representative subsample weighing about 4 pounds, and send under the inclosed frank by mail to my address.

H. W. Wiley, *Chemist.*

On March 20, 1901, the seed was mailed to the stations named by the Section of Seed and Plant Introduction, and on March 23 the following letter was addressed to the cooperating stations:

March 23, 1901.

Dear Sir: In order that I may be put into direct communication with the official of your station who will be in personal charge of the collaborative work on the study of the influence of environment on the sugar beet, I write to ask that in case you delegate this work to one of your assistants you inform me of that fact. In case you take personal charge of it please let me know also, in order that I may have an official record of the person immediately in charge.

---

[a] Results published in Bulletin No. 64, Bureau of Chemistry, U. S. Department of Agriculture, 1901.

H. W. Wiley, *Chemist.*

These letters were also later addressed to the Virginia station at Blacksburg. The responses showed that the following stations and officials would take part in the work:

Washington, D. C., G. L. Spencer; Lafayette, Ind., H. A. Huston; Agricultural College, Michigan, J. D. Towar; Ames, Iowa, James Atkinson; Lexington, Ky., M. A. Scovell; Geneva, N. Y., G. W. Churchill; Ithaca, N. Y., L. A. Clinton; Logan, Utah, John A. Widtsoe; Blacksburg, Va., W. B. Alwood; Madison, Wis., R. H. Shaw.

The work at the North Carolina station was temporarily abandoned, owing to a change in the personnel of the station.

On April 8, 1901, a final letter of instructions was sent out to the stations above-named, which read as follows:

April 8, 1901.

Dear Sir: I feel that it is scarcely necessary to make any suggestions in regard to the methods of planting and cultivating the beets which you undertake to grow in collaboration with this division. The seed, No. 5772 "special," you have probably already received. If not, please let me know at once.

Some time before sowing, preferably the previous autumn, the soil should be plowed to the usual depth of 8 or 9 inches, and subsoiled 6 inches deeper, making a seed bed at least 15 inches in depth. If the character of the soil warrants it, a deeper plowing, even to 10 or 11 inches, and a subsoiling of 6 inches additional will be advisable.

The surface of the soil should be reduced to a fine tilth, and be well harrowed and stirred immediately before planting, so as to stop all growth of weeds which may have been started.

The rows should be 18 inches apart, and the seed be planted at the rate of about 25 pounds per acre, so as to be sure of a good stand. If the soil be moist, the seed should be covered to a depth of from one-half to 1 inch. If the weather be dry, slightly deeper planting may be advisable.

So soon as the plants are growing vigorously they should be separated into clumps by a hoe 6 inches in width, leaving the length of 3 inches of beets in each hill. When the beets have a vigorous growth and begin to form the fourth leaf, they should be thinned to about one plant in each 9 inches. Where vacancies occur in a row, transplant carefully so as to have the number of plants indicated above.

Ordinary surface cultivation is all that is required, taking care not to cover up the beets at the first cultivation.

In sending the samples of soil, in accordance with previous instructions, do not forget to send a history of the plot, so far as known. Complete cultural and meteorological data in collaboration with the Weather Bureau should be kept and forwarded with the samples. Franks for forwarding samples and full instructions for harvesting and sampling will be sent later. It is earnestly requested that frequent analyses be made also at the station, so that the results of those analyses can be compared with those which are made of beets sent here.

Any questions in regard to further details will be cheerfully answered.

Respectfully,

H. W. Wiley, *Chemist.*

The following letter in regard to harvesting was forwarded to the cooperating stations under date of September 15:

> Dear Sir: Relative to the sugar beets grown by you from seed marked "No. 5772 special:"
>
> When the beets appear to be approaching maturity and before any second growth can take place, please harvest a sufficient quantity to enable you to make a fair estimate of the yield per acre. Select 30 average beets from those harvested, have the tops removed, leaving about an inch of stems, and wash and wipe the roots.
>
> Pack the carefully dried beets in a box, inclose full data relative to the sample on the slip "A," and forward the package to me by express, charges collect.
>
> I will forward you the necessary slips "A" and envelopes. Please repeat this sampling and the estimate of yield at intervals of a week until end of season, timing the shipments, if practicable, so that packages will not reach here Saturday or Sunday.
>
> Keep accurate data of all field work, which please transmit at end of season.
>
> Respectfully,
>
> H. W. Wiley, *Chief.*

Slip "A," referred to in the above letter, calls for the following data: Variety; when planted; when thinned; when harvested; date of shipment of sample; character of the soil; width between rows, inches; character of the growing season, favorable or unfavorable; estimated yield per acre, tons; remarks.

EXPERIMENTS CONDUCTED AT WASHINGTON, D. C.

Fourteen rows of the Dippe Kleinwanzlebener Elite beet seed (S. P. I. No. 5772) were sown on the experiment farm at the Potomac Flats on May 18. They showed above the ground on May 25, and were cultivated once a week and oftener when there were frequent rains. Between June 11 and 14 the beets were thinned, one-half being allowed to remain 8 inches apart and the other 12 inches apart. The distance between the rows was 18 inches. Hand hoes and rakes were used for the first cultivation, after which wheel hoes were employed. In the following tabulated data the thick stand and the thin stand of beets have been separately considered for purposes of comparison.

Agricultural and analytical data on beets grown on the experiment farm, Potomac Flats.

THICK STAND (PLANTS 8 INCHES APART).

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 26	33.4	6.6	8.3	7.4	....	66.7
October 3	31.6	6.2	7.6	7.3	7.1	65.8
October 10	34.1	6.7	7.9	7.8	7.5	68.4
October 17	33.5	5.9	8.1	8.0	7.8	67.2
October 24	33.0	6.7	8.1	8.6	8.4	65.6
October 31	22.5	8.6	11.8	9.4	9.1	69.1
November 7	34.0	6.7	5.1	8.8	8.5	67.7
November 21	34.2	5.1	7.0	10.6	10.3	68.8
Averages	32.0	6.6	8.0	8.5	8.4	67.4

Agricultural and analytical data on beets grown on the experiment farm, Potomac Flats—Continued.

THIN STAND (PLANTS 12 INCHES APART).

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 26	35.1	10.4	7.0	7.3	....	65.2
October 3	39.0	9.9	10.1	7.4	7.2	65.5
October 10	30.6	8.5	9.9	7.8	7.5	66.6
October 17	34.3	8.5	7.8	8.2	8.0	66.6
October 24	39.9	7.2	7.5	9.0	8.7	66.6
October 31	24.6	10.7	8.6	10.2	9.9	70.8
November 7	39.2	8.0	6.6	9.6	9.3	67.6
November 21	35.6	10.1	8.2	10.0	9.7	68.5
Averages	34.8	9.2	8.2	8.7	8.6	67.2
General averages	33.4	7.9	8.1	8.6	8.5	67.3

A comparison of the two plats represented by the tables under "thick stand" and "thin stand" shows that the crowding of the beets produced a marked effect upon the average weight, which in the "thick" plat was 6.6 ounces and in the "thin" plat 9.2 ounces, after topping. The term "topping" means the removal of the top of the beet at the neck, as in preparation for the factory. The beets were undersized on both plats, which is a matter of remark, inasmuch as the soil is composed of the deposit from the Potomac River bottom and is considered quite fertile, producing abundantly other crops grown in the same field. The yield per acre on the two plats was almost identical, the thin stand having a slight advantage. In regard to the yield of sugar the two plats are very close together, but contrary to expectation the small beets in the "thick stand" plat had slightly less sugar than the beets of larger growth. The purity in both plats was extremely low, but was slightly higher in the "thick stand" plat.

The meteorological conditions under which these beets were grown are shown in the following table:

Meteorological data for Washington, D. C., 1901.

Month.	Mean temperature.	Total precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	° F.	*Inches.*	*Hours.*	*Hours.*			
May	62.5	2.81	197.1	443.8	44	10	16
June	72.4	4.66	329.8	445.9	74	18	6
July	79.8	5.17	273.6	453.0	60	12	7
Averages and totals	71.6	12.64	....	....	59.3	40	29
August	76.0	4.12	267.3	423.2	63	11	9
September	67.4	1.61	245.4	373.4	66	11	5
October	55.6	0.97	268.0	346.0	77	19	6
Averages and totals	66.3	6.70	....	....	68.7	41	20
General averages and totals	69.0	19.34	....	....	64.0	81	49

The meteorological conditions were quite favorable to the growth of beets, especially in respect of the distribution of the rainfall. In the three months most important to growth, namely, June, July, and August, the rainfall was abundant, while in September, the month most favorable to ripening, and in October, the month of harvest, the rainfall was deficient. In respect of distribution of rainfall, therefore, the season was ideal for the growth of a sugar beet. The temperature of the three principal growing months, as was to be expected, was very much in excess of the maximum which is found to be best suited to the production of beets of high sugar content. The excess above the maximum of 70° F. for June was 2.4°, for July 9.8°, and for August 6°. The distribution of the clear and cloudy days was also favorable to the growth of the beets. Aside from the temperature, therefore, it may be said that the meteorological conditions under which these beets grew were extremely favorable. It would appear from the consideration of this single plat that the temperature is the most important factor in the production of a high-grade beet.

EXPERIMENTS CONDUCTED BY THE INDIANA STATION

Prof. H. A. Huston makes the following report as to the cultivation of the beets grown from the Dippe Kleinwanzlebener Elite seed at Lafayette:

> The special sugar-beet seed, No. 5772, furnished us by the Department in 1901, was planted on April 30, 1901. The land was plowed and subsoiled and was in fair condition at the time of planting. As two quite hot days preceded the planting, the soil temperature was sufficiently high. The seeds were planted in plats of six rows each, there being four plats. The rows were about 20 rods long, but the sampling was done upon the north end of the field because the conditions were more uniform there. The spaces between the four plats were filled out by three plats of 6 rows each from the other seed furnished by the Department. Samples of the soil and subsoil were drawn on May 1, 1901, and forwarded to the Department. The beets came up on May 9. The stand was not very good nor uniform, although double the usual amount of seed was used. As the season progressed the stand became still more uneven and was little improved by transplanting, although at two periods when conditions seemed to be favorable beets were transplanted to fill the vacancies. The beets were thinned to 8 inches in the row, and the rows were 22 inches apart. The cultivation was kept up into July. The season was decidedly unfavorable on account of the lack of moisture, and this fact combined with the rather uneven stand gave a low yield, although the beets were of very good quality. On November 1, the average yield from the four plats of No. 5772 was 5.4 tons of washed capped beets per acre. The weighings upon the other plats showed that the yields were not materially different from this. The last samples were drawn on November 1. On November 4, the ground froze and remained frozen for several days, and no further work was done upon the field, since the beets were badly frozen to a depth of 3 inches. Previous to the heavy freeze the bulk of the field had been harvested, a small portion only being left to study the question of ripening.

Agricultural and analytical data on beets grown at the Indiana experiment station, Lafayette.

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
	Per cent.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 20		6.3		12.1		85.2
October 5		6.2		15.5		93.3
October 26		7.7		15.5		91.7
October 28[a]	14	8.2		15.1	14.6	82.5
November 1		6.4	5.4	16.7		90.7
Average	14	7	5.4	15	14.6	88.7

[a] These analyses were made at Lafayette, with the exception of the one dated October 28, which was made at Washington from a sample corresponding to that analyzed on October 26 at the Indiana station.

The data for Lafayette show the growth of a beet very much undersized, the average size of the topped beet as prepared for the factory being only 7 ounces. The average yield per acre was also only about one-half the normal average--namely, 5.4 tons—though this figure is open to suspicion, only one estimate having been made. The percentage of sugar in the beets was quite high, and the purities were phenomenally high, as has been the case in previous reports. In the sample analyzed in the Bureau of Chemistry the purity was found to be 82.5, which leads us to believe that some modification of the ordinary method of ascertaining purity is used at the Lafayette station, which is the cause of the phenomenally high figures. In the platting of the curves in the charts which follow, the data used are those obtained in the Bureau of Chemistry on October 28.

The meteorological data for Lafayette and Indianapolis as given in the following tables illustrate clearly the conditions of drought and excessive sunshine that prevailed, although the actual average temperature was 1.2° lower than in 1900.

Meteorological data for Lafayette, Ind., 1901.

Month.	Mean temperature.	Precipitation.	Clear days.	Cloudy days.
	° F.	*Inches.*		
May	59.5	2.89	2	14
June	72.9	4.46	8	11
July	81.1	.44	18	2
Average and total	71.2	7.79	28	27
August	75.5	2.50	10	12
September	66.8	1.34	15	6
October	56.1	4.78	21	7
Average and total	66.1	8.62	46	25
General average and total	68.6	16.41	74	52

The sunshine record for Indianapolis, the nearest station at which sunshine records were kept, is shown in the following table:

Meteorological data for Indianapolis,[a] Ind., 1901.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	° *F.*	*Inches.*	*Hours.*	*Hours.*			
May	60.2	2.45	228.5	446.7	51	8	12
June	73.6	3.52	297.6	449.0	66	7	5
July	82.0	.83	396.3	455.2	87	19	0
Average and total	71.9	6.80	....	....	68	34	17
August	75.2	3.57	286.7	425.2	67	7	6
September	67.4	.66	284.1	373.6	76	14	2
October	56.7	3.52	247.8	344.9	72	17	7
Average and total	66.4	7.75	....	....	71.7	38	15
General average and total	69.2	14.55	....	....	69.85	72	32

[a] Fifty-nine miles southeast of Lafayette, Ind.

As will be seen from the preceding page the rainfall at Lafayette was very small, being only 16.41 inches as compared with 30.52 inches in 1900, and, moreover, the distribution was very unfavorable. In July, which is the principal growing month, there was scarcely any rain at all, while the supply for August was very moderate, and that for September still less. On the other hand, during October, the harvesting season, the rainfall was excessive. Thus it is seen that the distribution of the rainfall was not at all favorable to the production of the crop.

The temperatures for the growing season were above the maximum for the production of beets of the highest quality, being 2.9 degrees above 70° F. for June, 11.1 degrees above 70° F. for July, and 5.5 degrees above 70° F. for August. There is a remarkable contrast between the character of the beets grown at Lafayette and those at Washington, D. C., where it appears that the high temperature interfered very seriously with the production of sugar in the beet. In the case of Lafayette this effect does not appear, and the conclusion to be drawn from these data would be contrary to that reached at the Washington station. In this case it seems to be the latitude which is the predominating factor. The distribution of clear and cloudy days was very irregular, October having the largest number of clear days, although it was a month of heavy precipitation, July coming next and September third. The total number of clear days was 10 greater than in 1900, and the percentage of sunshine, as observed for Indianapolis, was 5.1 per cent higher.

The meteorological data for Indianapolis, about 59 miles southeast of Lafayette, are interesting for comparison. It is seen that the temperature and precipitation average about the same for the two points, but the distribution of the rainfall is slightly different, being heavier during August at Indianapolis and lighter during September. The total precipitation for the six months, however, was slightly greater at Lafayette, while the average temperature, as might be expected, was a little higher at Indianapolis.

Under date of October 28, Professor Huston, in commenting on the prevailing meteorological conditions during the beet season of 1901, says:

> As you are doubtless aware, the summer season has been quite unusual in this section, the drought being so severe that the corn crop will be reduced fully one-half; on our own farm we have practically no corn.
>
> Your circular of September 15, was received, but since the beets had not appeared to approach maturity, but were simply standing still from lack of water, I did not deem it wise to send samples at that time. On September 20, I sampled a field, and the results you will find on inclosed card; on October 5, I sampled again, and you will see that between these dates the beets had made a very substantial gain in both sugar content and purity. No rain fell between these dates. On October 11, a general rain set in, and in four days we had 4.35 inches of rain. The beets started to grow, but you will see from the results of the analyses on October 26, that the sugar content was not reduced. This is an unusual result, and from now on we shall sample the field every few days to see what takes place. The drought reduced the stand of beets fully one-half, but those remaining are of marketable size and very good quality.
>
> I regret very much that the beets are not so situated that we could have tried irrigation on them, for it would have been a banner year for the purpose.

EXPERIMENTS CONDUCTED BY THE IOWA STATION.

The beets were planted at the Iowa station on May 22, thinned on June 20, and harvested for the first time on October 7. Mr. James Atkinson, assistant in agriculture at Ames, writes as follows concerning the beet crop on September 23, 1901:

> I may say that, while our beet crop has run the gauntlet of many foes this year, still it looks exceedingly well at the present time. You are, no doubt, aware that our crop suffered considerably from the hot winds and drought during the summer. I found that the beet crop stood it about as well as any other crop, although it was not by any means exempt from injury. The blister beetles also gave us considerable trouble, and we were compelled to fight four broods of them with Paris green and London purple. However, it appears at the present time that we have come out on top and that we shall have beets of good quality to harvest. * * * Sugar making has scarcely commenced yet, the beets being only slightly sweet to the taste. I think the first harvesting will be ready about October 1.

The results of the analyses made at Washington of the three samples of beets forwarded are found in the following table:

Agricultural and analytical data on beets grown at the Iowa Experiment Station, Ames.

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
October 10	18.7	13.4	12.1	13.7	13.3	80.6
October 21	20.1	12.6	12.8	14.4	13.9	79.6
November 5	14	16.7	13.8	15.7	15.1	80.5
Average	17.6	14.2	12.9	14.6	14.1	80.2

The agricultural and analytical data for Iowa show beets of fine character, very favorable yield per acre, and satisfactory purity. The average weight of the beets, prepared for the factory, was 14.2 ounces and the estimated yield per acre 12.9 tons. The percentage of sugar in the beet was 14.1 and the purity 80.2. These data must be regarded as exceedingly favorable, especially in view of the climatic conditions under which the beets were grown.

The following detailed statement of the meteorological conditions shows the extent of the drought referred to by Mr. Atkinson, the rainfall for the six months being only 16.15 inches, as compared with 36.29 inches the preceding year. The temperature was practically the same, the record for 1900 showing an average of 68.2°.

Meteorological data for Ames, Iowa, 1901.

Month.	Temperature.	Precipitation.	Clear days.	Cloudy days.
1901.	°*F.*	*Inches.*		
May	61.4	3.69	17	5
June	74.4	2.36	21	1
July	83.3	2.26	27	1
Average and total	73.0	8.31	65	7
August	72.8	1.21	25	1
September	61.6	3.65	16	7
October	53.9	2.98	21	4
Average and total	62.8	7.84	62	12
General average and total	67.9	16.15	127	19

The sunshine record is taken from a table of meteorological data for Des Moines, Iowa, about 30 miles south of Ames, that being the nearest station at which sunshine records were kept:

Meteorological data for Des Moines, Iowa, 1901.

Month.	Temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1901.	°*F.*	*Inches.*					
May	61.6	1.40	312.7	451.9	69	12	6
June	73.4	2.41	329.1	456.2	72	11	3
July	84	1.72	390.1	461.8	84	15	1
Average and total	73	5.53	....	....	75	38	10
August	75	0.67	318.9	429.4	74	13	3
September	64.8	2.60	199.9	374.5	53	9	9
October	56.2	2.14	230	342.5	67	13	9
Average and total	65.3	5.41	....	....	64.7	35	21
General average and total	69.2	10.94	....	....	69.85	73	31

These data show that the months of June, July, and August were considerably above the desired temperature, the month of July especially having been excessively hot both at Ames and at Des Moines. The heat coupled with a very insufficient rainfall, July and

August showing a very small precipitation, made the season unusually dry. The data for Des Moines show a much smaller precipitation and at the same time a much smaller number of clear days than the data for Ames. These data show that the beet crop, properly planted and cultivated, is independent of variations in precipitation to a surprising extent. The total precipitation during the growing season at Ames for 1901 was considerably less than one-half that of the previous year and yet no deleterious effect was produced thereby on the size of the beets. In this respect and in quality the beet crop was markedly superior to that of 1900. These data are valuable as showing that, in regions where deficient rainfall may occur during the summer time, the sugar beet may produce a satisfactory crop if the proper attention is paid to the preparation of the soil and the cultivation.

EXPERIMENTS CONDUCTED BY THE KENTUCKY STATION.

The special beet seed was planted at Lexington, Ky., on April 29, 1901, 18 inches between the rows, thinned on May 23 and June 10, and harvested on October 17. The soil was plowed 8 inches deep and subsoiled 5 inches.

The analysis made at Washington of a sample of these beets gave the following data:

Agricultural and analytical data on beets grown at the Kentucky Experiment Station, Lexington.

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
October 21	25.2	10.4	8	9.3	9	71

The analytical and agricultural data obtained at Lexington during the season are as follows:

Agricultural and analytical data determined at the Kentucky station.

Date of sampling.	Number of beets.	Average weight topped.	Sugar in juice.	Purity coefficient.
1901.		*Ounces.*	*Per cent.*	
July 11	1	....	8.1	63.7
July 27	4	8	9.4	70.3
August 1	4	9.7	12.1	75.4
August 19	4	10.9	9.7	77.2
August 23	3	10.7	9.9	77.2
October 16	10	12.5	10.1	75.7
Average	....	10.4	9.9	73.3

The data for the Kentucky station show that the beets produced there were slightly under the normal size, and neither the content of sugar nor the purity was high enough to enable these beets to compete in sugar making with beets grown in more northern localities. The analysis of the single sample of beets made at Washington gave almost the same data as the average of the analyses made at the Kentucky station from July 11, to October 16, inclusive. From the analyses made at the Kentucky station it appears that the beets reached their highest content of sugar early in August, but too much stress must not be laid upon a single analysis. From the middle of August to the middle of October there was not a very large variation in the content of sugar in the beets.

Mr. Scovell states that the early season was favorable, but that July and August were unfavorable. The climatic conditions, as shown in the following table, do not differ greatly from those of the preceding season, even as to total rainfall:

Meteorological data for Lexington, Ky., 1901.

Month.	Mean temperature.	Total precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	Degrees.	*Inches.*	*Hours.*	*Hours.*			
May	62.8	2.67	273.4	441.7	62	14	7
June	73.9	3.70	322.3	443.1	73	13	2
July	80.3	2.61	385	450.1	86	23	0
Average and total	72.3	8.98	....	....	73.7	50	9
August	74	3.71	305.5	422.1	72	10	5
September	67	2.18	276	373	74	13	2
October	57.9	1.33	288.4	347.3	83	22	2
Average and total	66.3	7.25	....	....	76.3	45	9
General average and total	69.3	16.23	....	....	75	95	18

The meteorological data show that the temperature of June and July was lower than at Ames, Iowa, although Lexington is about 275 miles south of Ames. The distribution of the rainfall at Lexington was fairly favorable, although the supply was not sufficient. There is, however, a remarkable uniformity of precipitation during the months from May to September, inclusive. The precipitation for October was small, which is distinctly favorable for beet culture.

Mr. Scovell further calls attention to the existing drought by a statement of the accumulated deficiency of precipitation from January 1, 1901, which in May amounted to 8.12 inches; in June to 8.67 inches; in July to 10.24 inches; in August to 10.59 inches; in September to 11 inches, and in October to 11.78 inches.

A slight improvement took place in the quality of the beets, the average of sugar in the beet having been 7.8 per cent and the purity coefficient 69.6 in 1900, as compared with 9 and 71 for 1901. The beets are, however, still considerably below the commercial standard, both as to sugar content and purity.

EXPERIMENTS CONDUCTED BY THE MICHIGAN STATION.

As will be seen by reference to the following table, two sets of experiments were conducted at the Michigan station, field No. 3 having been planted May 16, with rows 18 inches apart, and thinned June 15, while field No. 6 was not planted until June 8, and was thinned July 8 and 9, the rows being 21 inches apart. The soil of field No. 3 is described as a sandy loam and that of field No. 6 as a gravelly loam. The constituents of the soil will be discussed in detail under that heading. While leaf spot checked the growth somewhat, the season was in general very favorable, cool, and with a great deal of sunshine. The data for both fields are as follows:

Agricultural and analytical data on beets grown at the Michigan Experiment Station, Agricultural College.

FIELD NO. 3, EARLY PLANTING, MAY 16, 1901.

When received.	Removed in topping.	Average weight after topping.	Yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 26	21.9	8.8	13.6	12.5	....	79.1
October 10	16	8.8	14.5	14.4	13.8	79.6
October 15	10.2	10.9	14.5	15.4	14.9	81.5
October 18[a]	14.2	8.2	16.9	14.1	13.7	80.1
November 2[a]	19.1	9.4	16.1	15.3	14.8	80.1
November 8	25.5	10.1	14.5	16.5	16	82.9
November 14	22.4	13.7	15.4	15.2	14.7	80.4
Average	18.5	10	15.1	14.8	14.7	80.5

FIELD NO. 6, LATE PLANTING, JUNE 8–9, 1901.

When received.	Removed in topping.	Average weight after topping.	Yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
September 26	15.1	5.4	5.3	12	11.7	79.5
October 10	12.5	6.4	5.4	14	13.4	89.9
October 15	11.8	4.1	5.4	14.8	14.3	82
October 18[a]	14.2	8.2	4.7	11.1	13.7	80.1
November 2[a]	19.1	9.4	5.1	15.3	14.8	80.1
November 8	24.2	6.4	4.6	17.4	16.6	83.7
November 14	23.7	7	6.6	16.8	16	82.4
Average	17.2	6.7	5.3	14.9	11.4	82.5
General average	17.9	8.3	10.2	14.9	11.6	81.5

[a] In the two packages arriving on these dates the beets harvested from the two fields could not be separated.

The analytical data furnished by the station showed an average weight of beets in the field of 13 ounces, 12.7 per cent of sugar in the beets, a coefficient of purity of 79.2, and a yield per acre of 11.6 tons.

In case of the early planting, while the average weight of the beets was slightly below the normal, the estimated yield per acre was far above the average, namely, 15.1 tons. The data for the late planting, June 8 and 9, field No. 6, show practically the same quality of beets as regards sugar content and an even higher purity, but, in so far as yield per acre is concerned, the crop was only about one-third that of the early planting.

The beets grown during 1901 were of a better quality than those of the preceding year, the percentage of sugar in the beet and the coefficient of purity being each 1.5 higher. The beets were smaller, however, and the average yield per acre was lower. The data for the late planting are responsible for the decrease to a great extent, the figures for the early planting being but little lower than those for 1900.

The climatic conditions existing at Agricultural College and at Detroit during this season are shown in the following tables:

Meteorological data for Agricultural College, Mich., 1901.

Month.	Temperature.	Precipitation.	Clear days.	Cloudy days.
	° *F.*	*Inches.*		
May	55.2	2.42	10	14
June	68.0	3.57	13	2
July	74.2	5.08	22	3
Average and total	65.8	11.07	45	19
August	68.4	2.49	15	7
September	61.7	1.67	18	7
October	49.6	4.61	18	4
Average and total	59.9	8.77	51	18
General average and total	62.8	19.84	96	37

In the following table is given the sunshine record for Detroit, Mich., 76 miles southeast of Agricultural College, the nearest point at which sunshine records were kept.

Meteorological data for Detroit, Mich., 1901.

Month.	Temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	° *F.*	*Inches.*	*Hours.*	*Hours.*			
May	56.6	2.76	222.2	451.9	49	5	10
June	68.8	2.08	286.5	456.2	63	9	8
July	76.8	5.50	333.4	461.8	72	18	3
Average and total	67.4	10.34	....	....	61.3	32	21
August	71.9	3.20	238.3	429.4	55	10	6
September	64.8	1.65	244.5	374.5	65	13	4
October	52.8	1.90	231	342.5	67	16	3
Average and total	63.2	6.75	....	....	62.3	39	13
General average and total	65.3	17.09	....	....	61.8	71	34

These data show an abundant rainfall during the three principal growing months, especially in July, when the amount appears to be excessive. September was a dry month, favorable to the ripening of the beet, while, on the other hand, October was a wet month and unfavorable to the harvesting. The figures for Detroit show practically the same conditions as those prevailing at Agricultural College. A comparison of the meteorological data with that for 1900 shows that there was a fall in the average temperature of 1.7 degrees and an increase in the precipitation of 2.3 inches, while the percentage of sunshine increased 2.6 per cent.

EXPERIMENTS CONDUCTED BY THE NEW YORK STATION AT GENEVA.

The experimental beet plots at the Geneva station were planted on June 10, with 20 inches between the rows, and were thinned from July 10 to 20. Special fertilizer experiments were conducted at this station, which will be further discussed under soil. The season was reported as being favorable, and the following analytical data, as determined at Washington, bear out this report, the beets from Geneva outranking any others grown as to sugar content and purity, standing second in size and yield.

Agricultural and analytical data on beets grown at the New York experiment station, Geneva.

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
November 2	16.8	19.7	....	16.5	16.0	82.1
Do	15.9	18.4	....	17.9	17.3	89.1
November 11	17.8	21.4	....	14.9	14.4	79.7
Do	17.8	19.7	....	15.8	15.3	83.2
December 24	88.3	10.8	....	15.45	....	82.6
Do	15.5	13.1	....	17.2	....	86.9
Averages	20.4	17.2	[a] 13.8	16.3	15.8	83.9

[a] Averaged from N. Y. Exp. Sta. Bul. No. 205, 1901.

As the conditions existing at Geneva seem to approach the ideal for sugar-beet culture, it is regretted that only the temperature and rainfall data are obtainable for that place. The sunshine data is not available for any nearer point than Ithaca. It is shown, however, that the decreased temperature in 1901, as compared with the previous year—i. e., 1.6°—and the increased rainfall of 3.4 inches had practically no effect on the quality of the beets, while their size was slightly increased. The meteorological data available are as follows:

Meteorological data for Geneva, N. Y., 1901.

Month.	Temperature.	Precipitation.
1901.	° *F.*	*Inches.*
May	56.9	3.80
June	68.9	2.07
July	76.6	3.97
Average and total	67.5	9.84
August	71	5.52
September	64	2.46
October	51.4	1.35
Average and total	62.1	9.33
General average and total	64.8	19.17

These data show a most favorable distribution of the rainfall during the growing season. There was not too much precipitation during May and June, the period of preparation. There was abundant precipitation during July and August, the period of rapid growth; a diminished precipitation during September, the period of ripening, and a very slight rainfall during October, the time of harvesting. No more ideal distribution of the rainfall could be desired.

EXPERIMENTS CONDUCTED BY THE CORNELL STATION AT ITHACA, N. Y.

The season at Ithaca is reported as having been favorable, and a comparison of the analytical data with that of the previous year shows that the beets were of practically the same high sugar content, although the purity was 2 points lower. While these beets are of excellent character, it is to be noted that the purity is not as high as would be expected in beets of their sugar content, being very slightly below the desired standard. The analyses made at the station give higher figures for both sugar content and purity than those obtained in our own work.

Agricultural and analytical data on beets grown at the Cornell experiment station, Ithaca.

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
October 17	17.9	12.2	10.1	14.5	14.1	80
October 24	17.3	11.2	13	14.5	14.1	81.9
October 31	15.8	13.7	13	14.5	14.1	77.5
November 7	21.6	13.3	13.5	16.3	15.8	81.1
November 21	26.7	11	13	14.6	14.2	79.3
November 25	15.8	17.3	13	15.8	15.3	79.4
Average	19.2	13.1	12.6	15	14.6	79.9

Agricultural and analytical data prepared at the Cornell Station.

When harvested.	Average weight of beets.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Ounces.*	*Per cent.*	*Per cent.*	
October 15	12.5	15.85	15.06	82.6
October 22	10.0	16.05	15.25	82.3
October 29	8.0	16.65	15.85	80.5
November 5	13.5	19.40	18.43	84.0
November 19	10.0	16.60	15.77	81.0
November 21	16.5	18.75	17.80	83.0
Average	11.75	17.22	16.35	82.2

The climatic conditions under which these beets were grown are shown in the following table:

Meteorological data for Ithaca, N. Y., 1902.

Month.	Mean temperature.	Total precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	°F.	*Inches.*	*Hours.*	*Hours.*			
May	55.8	4.20	219.6	451.9	49	6	16
June	67.8	3.06	344.7	456.2	76	10	8
July	74.3	3.60	369.4	461.8	80	5	8
Average and total	66.0	10.86	....	....	68.3	21	32
August	69.5	3.85	277.6	429.4	65	8	9
September	61.8	1.66	261.8	374.5	70	13	7
October	51.2	1.07	190.6	342.5	56	7	6
Average and total	60.8	6.58	....	....	63.7	28	22
General average and total	63.4	17.44	....	....	66	49	54

The meteorological data show an even distribution of rainfall, indicating, however, a larger precipitation in May and a smaller one in August than at the Geneva station. The dry weather of September and October was extremely favorable to the ripening and harvesting of the beets.

EXPERIMENTS CONDUCTED BY THE UTAH STATION.

The plat selected for the cooperative sugar-beet work was plowed and subsoiled on April 24, and seeded with a drill on April 27, 1901. The beets were thinned on June 1, and as late as June 19, were not suffering for water, at which time they stood 6½ inches. The dates of irrigation were July 1, 16, and 31; August 15; and September 3 and 16. The beets were cultivated on May 23, June 13, and July 3, the harvest taking place on November 4, 1901. The growth was uneven, there being vacant places in the rows. To the above report Director Widtsoe adds the following data, which differ somewhat from those obtained at the Washington office:

	Tons.
Yield per acre	23.91
Per cent sucrose in juice	17.05
Purity coefficient	81.0

The analytical and agricultural data obtained on the samples forwarded to Washington are as follows:

Agricultural and analytical data on beets grown at the Utah Experiment Station, Logan.

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
October 3	14.7	26.1	24.7	12.4	11.9	75.0
October 10	13.7	23.1	22.0	14.6	14.1	79.8
October 14	9.9	24.2	....	14.3	13.9	79.4
October 22	10.2	23.2	....	17.2	16.7	82.3
Average	12.1	24.2	23.4	14.6	14.2	79.1

The data for the Utah station are given for reference and comparison, but of course are not platted with the other stations in the graphic charts. It is the intention during another year to include a series of comparative tests at irrigation stations when the data obtained for Utah will serve as an introduction. The most marked characteristic of the Utah beets is their large size, being fully one-third above the ideal average for sugar beets of high quality. The yield per acre is also far above that obtained at the other stations where irrigation is not practiced. The sugar content of the beets is entirely satisfactory, although the purity, as might be expected from the character of the soil and the method of culture, is somewhat low.

The meteorological conditions prevailing at Logan and the vicinity during the period of growth were as follows:

Meteorological data for Logan, Utah, 1901.

Month.	Mean temperature.	Total precipitation.	Clear days.[a]	Cloudy days.[a]
	Degrees.	*Inches.*		
May	60.6	2.43	21	7
June	61.4	0.41	20	2
July	76.7	.07	28	2
Average and total	66.2	2.91	69	11
August	72.8	1.60	21	8
September	59.2	1.03	22	8
October	53.6	1.83	18	10
Average and total	61.9	4.46	61	26
General average and total	64.0	7.37	130	37

[a] Record made at Corinne, 19 miles southwest of Logan.

In the following table is given the sunshine record for Salt Lake City, Utah, the nearest point at which sunshine records were kept, 66 miles south of Logan.

Meteorological data for Salt Lake City, Utah, 1901.

Month.	Mean temperature.	Total precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	°F.	*Inches.*	*Hours.*	*Hours.*			
May	63.0	4.27	352.6	419.1	79	20	2
June	65.0	0.49	351.4	451.9	78	23	3
July	80.2	.31	386.0	458.6	84	19	2
Average and total	69.4	5.07	....	....	80.3	62	7
August	76.0	1.22	282.0	427.4	66	18	6
September	63.0	.66	302.9	374.0	81	21	3
October	55.6	.98	239.4	343.9	70	22	5
Average and total	64.9	2.86	....	....	72.3	61	14
General average and total	67.2	7.93	....	....	76.3	123	21

The meteorological conditions at Logan are interesting in showing the distribution of the precipitation and the variations in temperature. During July it is seen that only a trace of rain fell, while in August and October the precipitation compares favorably with that of some of the nonirrigated stations. The temperature at Logan during the growing season is decidedly lower than at the nonirrigated stations. July shows a high temperature, but June especially is low, and the August temperature is not much above 70°.

The data for Salt Lake City show practically the same distribution of precipitation as at Logan during the months of June, July, and August. The temperature, however, at Salt Lake was decidedly higher, especially during July, and the precipitation during the month of May was nearly twice as great as at Logan.

EXPERIMENTS CONDUCTED BY THE VIRGINIA STATION.

For the first time the experimental beet work was carried on at Blacksburg, in 1901, thus supplying a Southern station to take the place of North Carolina, where the work was temporarily discontinued.

The beets were planted on June 1, with 18 inches between the rows, thinned on July 5, and the first sample forwarded was harvested on October 12. The determinations of the percentage of sugar in the juice, as made at the experiment station, were as follows:

	Per cent.
September 20, 1901	6.65
September 30, 1901	8.95
October 12, 1901	12.62
October 22, 1901	12.69
October 31, 1901	12.77
Average	10.74

The analytical data obtained at Washington on the samples forwarded from Blacksburg and the climatic conditions prevailing during the period of growth are shown in the following tables:

Agricultural and analytical data on beets grown at the Virginia experiment station, Blacksburg.

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
October 15	8.7	5.6	10+	11.6	11.2	75.5
October 24	20.6	4	....	13.5	12.9	76.7
November 2	16	4	....	15.8	15.3	80.2
Average	15.1	4.5	10	13.6	13.1	77.6

Meteorological data for Blacksburg, Va., 1901.

Month.	Mean temperature.	Total precipitation.	Sunshine.[a]	Clear days.	Cloudy days.
	° F.	*Inches.*	*Per cent.*		
May	58.7	6.71	....	12	10
June	67.8	6.78	46.8	10	12
July	74	4.51	54.6	14	7
Average and total	66.8	18	50.7	36	29
August	68.6	10.53	40.8	7	12
September	61.6	2.59	61.3	15	9
October	52	.96	[b] 68	21	3
Average and total	60.7	14.08	56.7	43	24
General average and total	63.8	32.08	53.7	79	53

[a] These data were averaged from weekly means furnished by Mr. Alwood.
[b] To October 19 only.

In commenting on the season at Blacksburg, Mr. Alwood, under date of October 12, says:

> The weather conditions have been decidedly bad here this year, so that the crop is not extra heavy, but it can be considered a fair average. I think that at this time the beets are practically mature, but I fear that the wet season and consequent lack of sunshine will cause them to analyze very low in sugar. To the present time no second growth has begun; in fact, only a few of the bottom leaves have died. It is hardly probable that conditions as to sunlight will be such as to increase the sugar content very materially from this on.

It will be noticed that the general characteristics of the season at Blacksburg are in striking contrast to those at the other stations, unusual rains having prevailed at the former and drought at the latter. In forwarding the samples, Mr. Alwood said: "A more unfavorable year could not occur, but in this garden soil the result is fair."

A study of the agricultural and analytical data for Blacksburg shows a beet of phenomenally small size but of a fair average yield per acre. The sugar content of the samples was fair, but the purity was unsatisfactory. The high sugar content of this low latitude must be ascribed principally to the altitude of the station, which is about 2,100 feet. One of the principal objects in extending the collaborative work to Blacksburg was to determine the effect of altitude and its accompanying meteorological influences upon the sugar content of the beet. This object has been attained in a most striking manner in the preceding data.

The rainfall during the preparatory planting period was excessive. For the month of June it was abundant and for August far in excess of the quantity required for favorable growth. The precipitation for September and October, however, was favorable to the ripening and harvesting of the crop.

EXPERIMENTS CONDUCTED BY THE WISCONSIN STATION.

While the beets grown at the Wisconsin station were of very fair quality, they were far inferior to those of the previous season, showing a decrease of sugar in the beet of 2.5 per cent and a decrease in purity of 8.8. The beets grown in 1900 ranked next to the highest among the cooperating stations, and undoubtedly the falling off in quality in 1901 was due not solely to the drought (the total precipitation for the growing season being 7.7 inches less than in the previous year), but also in part to the unfortunate distribution of such rain as fell. The drought was most extreme in the first months of growth and the rainfall greatest in September and October, thus probably inducing a second growth and lowering the quality of the beets. The average temperature and the number of clear and cloudy days remained practically the same as in 1900, the rainfall being thus the only important variant.

The tables given below show the analytical data obtained at Washington on these samples and also the meteorological conditions under which the beets were grown:

Agricultural and analytical data on beets grown at the Wisconsin Experiment Station, Madison.

When received.	Removed in topping.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beets.	Purity coefficient.
1901.	*Per cent.*	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
October 1	10	8.2	11.375	13.5	12.9	80.3
October 9	15.1	8.3	9.3	14.1	13.5	82
October 16	9.4	11.4	12.8	11.1	10.8	71.2
October 24	14.9	15.8	10.5	13.8	13.4	76.2
Average	12.4	10.9	11	13.1	12.7	77.4

Meteorological data for Madison, Wis., 1901.

Month.	Mean temperature.	Total precipitation.	Clear days.	Cloudy days.
	° F.	*Inches.*		
May	57.8	2.41	4	15
June	70.4	2.40	5	12
July	79.6	1.54	12	9
Average and total	69.3	6.35	21	36
August	71.6	1.33	10	11
September	61.4	4.16	7	10
October	52.4	2.49	10	9
Average and total	61.8	7.98	27	30
General average and total	65.6	14.33	48	66

The following comment on the climatic conditions as shown in the above table is made by Mr. Shaw:

The season of 1901 was, as a whole, very unfavorable to most crops in Wisconsin. The extremely hot and dry weather during July and August did great damage to crops all over the State, the southern part of the State suffering most from the drought. The average temperature of July was 4.8° F. above normal, and in the region along the western border of the State the average for this month rose 10° F. above the normal temperature. The rainfall during the season was very uneven, the average for the northern and central counties being nearly 23 inches for the seven months, March to September, and only about 14 inches for the southern counties. The total rainfall for the seven months was, on the average for the whole State, 20.41 inches, which is two-thirds of an inch below normal.

The following detailed report of the cultural data and other interesting items as to the season's work was forwarded by Messrs. Woll and Shaw, in charge of the cooperative work at the Wisconsin station:

The land was plowed early in the spring of 1901 and a few weeks later prepared in the usual manner for the planting of the beet seed by disking and pulverizing. The planting was done by means of a hand seeder on May 17, in rows running north and south, 18 inches apart. The experience of last year was duplicated this spring; a hard crust was formed on the land through heavy rains a few days before the young plants began showing themselves above ground; as most of the young plants probably would have been unable to break the crust formed, it was decided to reharrow and replant the entire plat, and this was accordingly done on June 7.

The plants began appearing above the ground about June 14. A terrific thunderstorm appearing on June 16 did great damage to the beets, as to all crops in this vicinity, and the soil of the eastern part of the field was washed badly, in places to a depth of 2 or 3 inches; in other places the young beet plants were covered to a similar depth. The field was thoroughly hoed, and although the crop at first seemed entirely ruined, the beet plants gradually recovered. In the eastern part they seemed struggling for existence against heavy odds for a week or more.

The plants were thinned from June 27 to July 1, a strong plant being left every 9 inches in the row. Owing to the severe drought, no transplanting was attempted. For over a month after this date no rain fell and the beets grew but little, the best stand being in the southwest corner and the poorest in the southeast corner of the field. The drought in this vicinity completely ruined some crops, and the prospects were at this time that there would not be a yield of 50 per cent of the usual average of any crop.

The sampling of the beets for analysis took place for the first time September 25, and from that time up to harvest the variety No. 5772 was sampled every week. The results of the analysis are given in the following table:

Agricultural and analytical data determined at Madison, Wis., on beets grown at that station.

Date of sampling.	Weight of trimmed beets.	Sugar in juice.	Sugar in beets.	Purity coefficient.
1901.	*Ounces.*	*Per cent.*	*Per cent.*	
September 25	9.6	12.6	12.0	77.2
October 4	8.0	13.7	13.0	78.4
October 11	10.7	10.8	10.2	71.6
Average	9.4	12.4	11.7	75.7

The sampling of No. 5772 was done by digging all beets in 60 feet of a row of average luxuriance and selecting three beets from the lot. Samples were forwarded to the Bureau of Chemistry, United States Department of Agriculture. The low results of analyses were due to the immature condition of the beets. A rainy period set in on October 7, which lasted for five days; over 2 inches of rain fell during this time.

The weather was raw and cold during the following days, and the harvesting was postponed as long as practicable, so as to give the beets a chance to mature. Other farm work and the uncertainty of the advanced season rendered it necessary, however, to begin the harvest on October 18. At that time the beets looked as if they were still growing; but few dead leaves were observed, and the general appearance of the beets was green and thrifty. The beets on the western half of the plat looked much better than those on the eastern half.

A bushel basket of beets was taken from each load hauled off the field (1 to 2 bushels of each variety). The samples thus secured were washed to determine shrinkage from adhering dirt. This amounted to 8.8 per cent for some of the different varieties, and on the average to 5 per cent. The results obtained at harvest were as follows:

Estimated yield per acre	tons	9.9
Sugar in the beets	per cent	10.9

The low results obtained during the past season with the university farm beets were somewhat of a surprise to us, although we did not expect much this year from the adverse conditions under which the beets grew throughout the season, the most important of which was the late date of planting. The peculiar climatic conditions, together with the short growing period which the beets had, fully explain the results, and also show that soils which in ordinary seasons will produce rich beets, higher in sugar than the common factory standard by at least several per cent, as has been generally the case on our university farm soil, may with the same kind of careful culture under exceptionally unfavorable circumstances produce beets that would not be accepted at a sugar factory.

The results of the analyses of beets grown by Wisconsin farmers during this season show that similar conditions did not prevail in all portions of the State. From three to four weeks after the beets were harvested the weather was most favorable to the maturing of beets, being sunshiny and quite warm, and, if the harvesting could have been postponed to this period, there can be no doubt that the results would have been nearly up to the standard set by earlier work in this line done at our experiment station. This would, however, have brought the harvest nearly a month and a half later than usual, which under ordinary fall conditions would be impracticable or at least quite inconvenient. The possible improvement of beets during the weeks following the harvest is suggested both by comparison with analyses of beets received from outside points before and after the time of our beet harvest and from what we know of the relations of weather conditions to the quality of the beets grown.

THE SOILS.

For the first time in the study of the effect of environment on the composition of the beet we have collected and examined a number of the soils on which the experiments were conducted. Unfortunately data on some soils are wanting, owing to the failure of the collaborating station to forward samples. The following data are therefore given tentatively, and in so far as possible the influence of the soil on the composition of the beets and the magnitude of the crop has been studied. The notes descriptive of the soils received from the stations follow on the next page.

DESCRIPTIVE NOTES ON SOILS.

Lafayette, Ind.

(Nos. 22383 and 22384.)

The samples of soil received from the Indiana station were accompanied by the following description:

I send you to-day a sample of the soil and a sample of the subsoil from the land on which the work on sugar beets will be conducted. This land has not been used for experimental plats, but has been in bulk crops, following the general rotation of corn, oats, wheat, and clover. Before the last corn crop I think there was millet on it. Last year the land was in clover with a poor stand, so that the beets are planted upon a clover sod which consists largely of weeds. The land is in the northwest corner of the field immediately west of the station building and is opposite the old greenhouse.

Agricultural College, Mich.

Field No. 3, Nos. 23581 and 23582.
Field No. 6, Nos. 23583 and 23584.

Under date of November 11, 1901, Mr. J. D. Towar, agriculturist of the station, made the following report on the soil on which the beets were grown:

In this mail I am sending you samples of the soil and subsoil on which the sugar beets which we have been sending you from seed No. 5772 were grown. The sample marked field No. 3 was a clover sod of two years' standing which was covered during the winter uniformly with a coat of stable manure. As soon as we could work the ground in the spring it was plowed to a depth of 8 inches and subsoiled 7 inches deeper. This operation was immediately followed by the roller, and the ground was harrowed at frequent intervals until May 9, when it was in fine condition, and an application of 200 pounds of home-mixed fertilizer per acre was made. This fertilizer consisted of one part nitrate of soda, one part muriate of potash, and two parts dissolved phosphate rock, the latter giving an analysis of about 17 per cent total phosphoric acid. The nitrate of soda was 96 per cent pure, and the muriate of potash contained 49.85 per cent K_2O.

The soil samples were taken August 28. In field No. 3 four samples of the soil were taken in the following manner, where the depths were respectively 9 inches, 8½ inches, 6½ inches, and 11½ inches. In each case a hole was dug about 1 foot square, leaving one perpendicular side from which a vertical slice about 3 inches in thickness was taken. The several samples were thoroughly mixed, and the sample sent is a portion of this mixture. The samples of subsoil, taken immediately below the soil samples, were from a depth of 1 foot, and were procured in a similar manner.

The samples from field No. 6 were taken in a similar manner to those from field No. 3, though the soil, a heavier loam, was from a plot which has grown sugar beets three years in succession, receiving absolutely no fertilizer. The depth of soil in the three places sampled was 8½, 8, and 9¼ inches. This plot was adjacent to others which have received each year applications of fertilizers, but the remarkably low yield of the No. 6 plot is due more to the lateness of the season at which the seed was planted than to exhaustion of the soil fertility. I wrote you some time ago that we had sown this seed on a piece of muck land, some of which was quite thoroughly mixed with alluvial soil.

Logan, Utah.

(No. 23586.)

The history of the plot used for the special beet work at the Utah station was reported as follows:

Plot No. 379 is located on the upper level of the Logan delta. The soil is gravelly and not more than 2 feet in depth. It is underlaid by a stratum of coarse porous gravel perhaps 25 feet or more in thickness. The virgin ground was broken in 1889. Corn was grown on it in 1890 and 1891, oats in 1892, clover in 1893, wheat in 1894, timothy in 1895 and 1896, wheat in 1897, peas in 1898, wheat in 1899 and 1900, and sugar beets, United States Department of Agriculture No. 5772 "special," in 1901. The plat was manured in the winter of 1900–1901.

Blacksburg, Va.

(No. 23818.)

The soil in which the beets were raised is described as a brownish-black loam. The following description of the taking of the sample forwarded to Washington for analysis and its general characteristics was furnished by the director of the station:

Three positions, fairly representing the entire plot, were chosen, and the notes made as follows:

Opening No. 1.—The soil down to the subsoil measured 7¼ inches, dark brown color, mellow loam in character. Color changed sharply at this point. Subsoil ocherous gray, quite friable, with considerable sand, yet firm.

Opening No. 2.—Same general character of soil; 9 inches down to the line of change of color.

Opening No. 3.—Soil 8½ inches to the line of change of color. The loam is a lighter brown in color. The subsoil is the same.

This land is a part of the experimental garden which was heavily manured until the last three years, during which time it had received no barnyard manure and no chemical fertilizers. It would be considered a very good type of garden soil, but in its present state is not very rich, as it has been cropped heavily each year. Last year on this area we grew a collection of Chinese vegetables, chiefly root crops, but no beets or crops belonging to this family.

Madison, Wis.

(Nos. 25051 and 25052.)

The history of the soil on which the sugar beets of 1901 were grown is as follows:

The plat set apart for the sugar beets at the university farm was a piece of land one-half acre in area (155 by 141 feet) in the northwestern portion of the Randall field. The field has been used as a pasture ever since this region was settled, and was in corn last year. During late years, prior to 1900, it served as a pasture for sheep or cows, but has never been otherwise manured. The soil is a clay loam, and, like most of the land on the university farm, has a tendency to bake after rains. The land produced a very good corn crop last year, and, as regards its state of fertility, should have been well adapted to the production of sugar beets. The lower part of the field slopes toward the northeast, and the unevenness in the soil in different parts of the field thus introduced rendered it somewhat unsatisfactory for variety tests.

Ithaca, N. Y.

(No. 25099.)

Unfortunately the sample of soil was not taken at Ithaca until 1902, and in the meanwhile a crop of oats and a crop of hay from clover and timothy had been harvested. The analysis of the sample of soil from the plat on which the beets were grown in 1901 is, however, submitted with the above explanation.

Ames, Iowa.

(No. 25114.)

The sample of soil from the plat on which the beets were grown in 1901 was not taken until 1902, and Mr. Atkinson, writing under date of October 28, 1902, in regard to this soil, says:

> I may say that there was a crop of soy beans grown on this plat during 1902, but I have had samples of the soil of the first 6 inches, the second 6 inches, and the third 6 inches taken, and will forward them to you if you desire to have them now. Of course I understand that the soy beans will have produced a very marked effect on the soil, particularly on the amount of nitrogen present. However, I shall dry the samples and forward them to you, and hope that they may be of some use in compiling results.

The samples sent were so small that the three were mixed and one analysis made.

Potomac Flats, Washington, D. C.

(No. 25125.)

This soil is an alluvial silt pumped from the Washington harbor side of the Potomac River. It was first plowed in 1898, and has never been fertilized. In 1899 the plat lay fallow, and in 1900 it was in corn.

Lexington, Ky.

No sample was forwarded from this station, but the soil in which the experiments were conducted is described as the clay loam of the station farm known as bluegrass soil.

Geneva, N. Y.

No sample of soil was received from Geneva, but the following descriptive data were furnished, relating to the fertilizer experiments: The general character of the soil was that of a clay loam. Comparative experiments were carried on with farm manure and commercial fertilizer, and for the first time in four years the beets raised on the plats to which the commercial fertilizer had been applied were superior to those on which stable manure had been applied.[a] This is attributed to the fact that an excessive amount of the latter was used. The analytical data obtained at Geneva bearing on this point is shown in the following table:

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[a] For a full discussion of the work from this point of view see Bul. No. 205, N. Y. Exp. Sta., Influence of Manure on Sugar Beets, December, 1901.

Agricultural and analytical data prepared at the New York Experiment Station, Geneva.

Fertilizer.	Quantity of fertilizer per acre.	Beets grown.				
		Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beets.	Purity coefficient.
	Pounds.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
Stable manure	80,000	13.1	14.9	18.6	13.4	80.0
Commercial fertilizer	1,000	12.0	12.6	20.7	15.6	87.7
Average	....	12.7	13.8	19.7	14.5	83.4

ANALYSES OF SOILS.

In the comparative table of analyses given below, including such stations as sent samples of soil, two methods of examination were employed, namely, the method of the Association of Official Agricultural Chemists,[a] and the method of ascertaining the quantity of mineral matter soluble in N/200 hydrochloric acid, as proposed by C. C. Moore, of the Bureau of Chemistry.[b] The first method gives practically all of the mineral matter in the soil that may become available in many years. The second method represents an attempt to determine the quantity of mineral matter (in this case potash and phosphoric acid only) which is available for the immediate uses of a crop. The method has been developed, however, with special reference to the oat plant. A glance at the analytical data obtained shows a wide difference in the character of these soils, both in respect of the total amount of plant food eventually available and the amount immediately available.

Chemical analyses of sugar-beet soils, 1901.

Station.	Soil sample.		Insoluble matter.	Water (H_2O).	Volatile.	Nitrogen (N).	Soluble in N/200 hydrochloric acid.	
	Serial number.	Description.					Potash (K_2O).	Phosphoric acid (P_2O_5).
			Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Lafayette, Ind.	22383	Soil	79.40	1.60	8.83	0.295	.0074	.00017
	22384	Subsoil	78.74	1.92	8.00	.239	.0051	.00014
Agricultural College, Mich.	23581	Soil (field No. 3)	91.87	.28	3.62	.154	.0036	.000062
	23582	Subsoil (field No. 3)	92.03	.24	2.44	.091	.0102	.00017
	23583	Soil (field No. 6)	91.87	.46	4.30	.161	.0052	.00009
	23584	Subsoil (field No. 6)	93.59	.26	2.08	.070	.0039	.00015
Logan, Utah	23586	Soil	71.54	1.26	10.75	.201	.0333	.00143
Blacksburg, Va.	23818	....	89.00	.42	4.46	.154	.0198	.00003
Madison, Wis.	25051	Soil	82.77	6.50	4.45	.165	.0040	.00040
	25052	Subsoil	79.81	5.20	4.40	.105	.0027	.000025
Ithaca, N. Y	25099	Soil	78.86	8.24	5.50	.201	.0077	.00003
Ames, Iowa	25114	.....do	78.49	2.82	8.45	.267	.0079	.00020
Washington, D. C.	25125	.....do	83.47	1.75	5.35	.183	.0062	.00020

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[a] Methods of Analysis, Bul. 46, Revised, Bureau of Chemistry, U. S. Dept. Agr.

[b] Journal American Chemical Society, *24*, 79.

Chemical analyses of sugar-beet soils, 1901—Continued.

Station.	Soil sample.		Soluble in 1.115 sp. gr. hydrochloric acid.					
	Serial number.	Description.	Sulphuric acid (SO_3).	Potash (K_2O).	Lime (C_aO).	Magnesia (MgO).	Fe_2O_3 Al_2O_3 Mn_3O_4.	Phosphoric acid (P_2O_5).
			Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Lafayette, Ind	22383	Soil	0.09	0.85	0.60	0.60	8.49	0.12
	22884	Subsoil	.06	.36	.72	.62	9.83	.10
Agricultural College, Mich.	23581	Soil (field No. 3)	.07	.12	.46	.32	3.33	.06
	23582	Subsoil (field No. 3)	.09	.18	.43	.45	4.60	.03
	23583	Soil (field No. 6)	.06	.06	.34	.22	3.30	.06
	23584	Subsoil (field No. 6)	.03	.13	.32	.35	3.61	.04
Logan, Utah	23586	Soil	.07	.76	5.00	3.42	7.49	.24
Blacksburg, Va	23818		.07	.80	.14	.29	5.57	.10
Madison, Wis	25051	Soil		.35	.87	.59	4.42	.04
	25052	Subsoil		.38	.52	.75	9.82	.06
Ithaca, N. Y	25099	Soil	.09	.22	.16	.56	6.55	.13
Ames, Iowa	25114	do		.35	.85	.65	8.22	.06
Washington, D. C	25125	do		.39	.47	.51	8.27	.03

Mechanical analyses of sugar-beet soils, 1901.[a]

Station.	Soil sample.		Fine earth.							
	Serial number.	Description.	Organic matter.[b]	Gravel, 2 to 1 mm.	Coarse sand, 1 to 0.5 mm.	Medium sand, 0.5 to 0.25 mm.	Fine sand, 0.25 to 0.1 mm.	Very fine sand, 0.1 to 0.05 mm.	Silt, 0.05 to 0.005 mm.	Clay, 0.005 to 0.0001 mm.
			P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
Lafayette, Ind	22383	Soil	3.52	1.94	4.00	2.22	2.00	5.76	62.08	22.00
	22384	Subsoil	3.00	1.37	3.12	2.06	1.72	5.35	58.23	18.15
Agricultural College, Mich.	23581	Soil (field No. 3).	1.62	2.84	6.76	12.20	34.54	15.84	18.22	8.86
	23582	Subsoil (field No. 3).	.54	2.78	5.32	11.60	37.60	14.96	13.26	13.86
	23583	Soil (field No. 6).	1.73	5.18	7.58	14.86	41.76	9.64	13.70	7.24
	23584	Subsoil (field No. 6).	.80	4.84	6.80	14.58	44.06	10.66	11.04	7.12
Logan, Utah	23586	Soil	3.24	.62	1.90	1.84	15.48	27.62	25.26	26.62
Blacksburg, Va	23818	do	2.07	2.68	3.84	4.68	9.62	12.66	48.10	18.00
Madison, Wis	25051	do	2.41	.48	1.46	2.26	6.36	11.82	64.12	13.36
	25052	Subsoil	.68		.46	.94	3.68	10.52	69.46	14.76
Ithaca, N. Y	25099	Soil	2.09	6.10	4.78	3.74	10.30	16.26	38.88	19.98
Washington, D.C.	25125	Soil (from flats).	1.63	.34	1.52	2.94	16.78	24.00	33.02	21.40

[a] Made in the Bureau of Soils, U. S. Department of Agriculture.
[b] Organic matter determinations were made by the wet combustion method.

COMMENT ON ANALYSES.

The study of the analytical data in the above tables is interesting from a scientific point of view, and is also conclusive, in so far as one series of observations can be, in regard to the very small effect which the composition of the soil has upon the sugar content of the beet. While it is doubtless true that the character of the soil influences to a greater or less degree the quality of some crops, it is certain that its principal influence in the case of the sugar beet is exerted almost exclusively upon the magnitude of the crop. In this connection the writer would like to recall from personal experience two instances showing how widely different kinds of soil may produce beets which have practically the same content of sugar. A few years ago samples of beets were received from Chautauqua County, N. Y., which were grown in a reclaimed swamp where the drainage had been so perfected as to permit the cultivation of the soil. The beets grown in this soil, extremely rich in vegetable mold, had a very high content of sugar. On the other hand, samples of beets taken from almost a pure sand near the Kankakee River in Indiana, where there was scarcely any organic matter in the soil, had almost the same content of sugar. These two types of soil were as entirely different as can well be imagined.

RELATION OF CROP TO THE PHYSICAL PROPERTIES OF THE SOIL.

Among the physical properties of the soils in question, determined for us by the Bureau of Soils of this Department, the first to be considered is the content of clay. The soil having the lowest amount of clay was that from field No. 6, Agricultural College, Mich., 7.24 per cent. Low clay content is usually associated with a high percentage of sand, and such is the case in this instance, the total sand of all dimensions being nearly 75 per cent. The highest clay content is found in the sample from Utah, namely, 26.62 per cent. This, of course, would indicate a low percentage of sand, which, in point of fact, is only about 45 per cent. The percentages of clay in the three soils producing the beets with the highest content of sugar (about 14.6 per cent in each case) were as follows: In the sample from Agricultural College, Mich. (field 3), 8.86 per cent; in that from Ithaca, N. Y., 19.98 per cent, and in that from Lafayette, Ind., 22 per cent, while the soil producing the poorest beets, namely, Washington, D. C., had 21.4 per cent of clay. It is evident from a study of the figures grouped in the following table that, while the texture of the soil, as shown by the mechanical analysis, undoubtedly has a direct bearing on the yield per acre, it has practically no effect on the content of sugar in the beet:

Mechanical analyses of soils and data regarding the crops of sugar beets grown thereon.

Serial number.	Station.	Sugar-beet crop.		Mechanical analyses of soils.		
		Sugar content.	Yield per acre.	Clay.	Silt.	Total sand.
		Per cent.	*Tons.*	*Per cent.*	*Per cent.*	*Per cent.*
25125	Washington	8.5	8.1	21.40	33.02	45.24
25051	Madison, Wis	12.7	11	13.36	64.12	21.90
22818	Blacksburg, Va	13.1	10	18	48.10	30.80
22283	Lafayette, Ind	14.6	[a] 5.4	22	62.08	13.98
25099	Ithaca, N. Y	14.6	12.6	19.98	38.88	35.08
23581	Agricultural College, Mich.[b]	14.7	15.1	8.86	18.22	69.34

[a] Only one estimate.

[b] Field No. 3.

RELATION OF CROP TO THE CHEMICAL PROPERTIES OF THE SOIL.

In considering the effect of the chemical composition of the soil upon the character and magnitude of the crop we have a problem of intricate difficulties. If we regard the soil in the light of the total plant food contained therein as indicated by treatment with hot concentrated hydrochloric acid for a considerable period of time we introduce figures which must be foreign to the problem in question. In point of fact, in order to have any accurate conception of this problem it is necessary to differentiate the amount of any given plant food consumed by a given crop from the total supply which is present. This might be illustrated by an attempt to determine how much nourishment would be given a man of average size during a day by analyzing the total nourishment in a barrel of flour from which his day's supply of bread has been made. The actual effect produced upon the man would not be represented by the total amount of flour in the barrel, but only by the total amount of flour he consumed. Therefore, as before stated, in considering the influence of the composition of the soil upon that of the beet and the magnitude of the crop it is important to know first the total quantity of plant food present, and next to ascertain if possible what portions are immediately available for the use of the crop. The following table has been arranged in order that the interrelations of these factors may be more conveniently observed:

Chemical analyses of soils and data regarding the crops of sugar beets grown thereon.

Serial number.	Station.	Sugar-beet crop.		Chemical analyses of soils.				
		Sugar content.	Yield per acre.	Nitrogen.	Potash.		Phosphoric acid.	
					Total.	Available.	Total.	Available.
		Per cent.	*Tons.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
25125...	Washington..........	8.5	8.1	0.183	0.39	0.0062	0.03	0.00020
25051...	Madison, Wis........	12.7	11.0	.165	.35	.0040	.04	.00040
23818...	Blacksburg, Va......	13.1	10.0	.154	.80	.0198	.10	.00003
22383...	Lafayette, Ind.......	14.6	[a] 5.4	.295	.35	.0074	.12	.00017
25099...	Ithaca, N. Y..........	14.6	12.6	.201	.22	.0077	.13	.00003
23581...	Agricultural College, Mich.[b]	14.7	15.1	.154	.12	.0036	.06	.00062

[a] One estimate only.

[b] Field No. 3.

Our studies of this problem are too restricted to justify any conclusion at present further than to call attention again to the patent fact that the quality of this crop is affected only in a minor degree by the chemical and physical properties of the soil. The case is quite different in respect to the kind of crop best suited to a given soil and the magnitude of the harvest. These two items are intimately associated both with the texture of the soil and the quantity of plant food therein.

SUMMARY OF DATA.

Summary of agricultural and analytical data, 1901.

Station.	Mean weight of topped beets.	Estimated yield per acre.	Sugar in beet.	Coefficient of purity.
	Ounces.	*Tons.*	*Per cent.*	
Washington, D. C	7.9	8.1	8.5	67.3
Lexington, Ky.[a]	10.4	8	9	71
Madison, Wis	10.9	11	12.7	77.4
Blacksburg, Va	4.5	10	13.1	77.6
Ames, Iowa	14.2	12.9	14.1	80.2
Logan, Utah	24.2	23.4	14.2	79.1
Agricultural College, Mich	[b]8.3	[b]10.2	14.6	81.5
Lafayette, Ind.[a]	8.2	[c]5.4	14.6	82.5
Ithaca, N. Y	13.1	12.6	14.6	79.9
Geneva, N. Y	17.2	13.8	15.8	83.9

[a] One sample analyzed at Washington.
[b] Average of late and early planting; data for early planting gives average weight 10 ounces, and average yield 15.1 tons.
[c] One estimate, for November 1, 1901.

Summary of meteorological data, May to October, 1901.

Station.	Mean temperature.	Precipitation.	Clear days.	Cloudy days.	Sunshine.
	°*F.*	*Inches.*			*Per cent.*
Washington, D. C	69	19.34	81	49	64
Lexington, Ky	69.3	16.23	95	18	75
Madison, Wis	65.6	14.33	48	66	....
Blacksburg, Va	63.8	32.08	79	53	[a]53.7
Ames, Iowa	67.9	16.15	127	19	[b]69.85
Logan, Utah	64	7.37	130	37	[c]76.3
Agricultural College, Mich	62.8	19.84	96	37	[d]61.8
Lafayette, Ind	68.6	16.41	74	52	[e]69.85
Ithaca, N. Y	63.4	17.44	49	54	66
Geneva, N. Y	65.5	18.03	See Ithaca.		

[a] Average of weekly data furnished by Blacksburg station.
[b] Observed at Des Moines.
[c] Observed at Salt Lake.
[d] Sunshine record for Detroit.
[e] Observed at Indianapolis.

Summary of geodetic data for experiment stations.

Station.	Average length of day.[a]		Latitude.[b]			Altitude.[b]
	h.	*m.*	°	′	″	*Feet.*
Washington, D. C	14	23	38	53	23	37.5
Lexington, Ky	14	18	38	02	25	979
Madison, Wis	14	44	43	04	36	955
Blacksburg, Va	14	14	37	14	00	2,100
Ames, Iowa	14	38	42	02	00	917
Logan, Utah	14	37	41	44	00	4,506
Agricultural College, Mich.[c]	14	42	42	45	00	847
Lafayette, Ind	14	30	40	23	00	542
Ithaca, N. Y	14	41	42	27	00	810
Geneva, N. Y	14	44	42	53	00	453

[a] These figures are for May to August, inclusive, and are from the records of the U. S. Naval Observatory.
[b] Data furnished by the U. S. Coast and Geodetic Survey.
[c] Determinations for Lansing, Mich.

CONCLUSIONS.

As in the previous report on this subject, the data obtained in the studies described in the previous pages are for convenience platted graphically in three charts, figs. 1, 2, and 3.

Chart No. 1 shows the percentage of sugar in the beet, the latitude of the station, and the sunshine record, including the total percentage and its distribution. The plat is based upon the percentage of sugar in the beets, beginning with the station having the lowest record and ending with that having the highest. Since North Carolina has dropped out of the list of stations cooperating, Washington enjoys the distinction of having produced beets with the lowest content of sugar. In order of sugar content, the other stations are arranged in an ascending scale, as follows: Lexington, Madison, Blacksburg, Ames, Agricultural College, Lafayette, Ithaca, and Geneva.

In general it may be seen that the latitude, as in the first year's experiments, follows the sugar content. A notable exception to this is found in the case of Blacksburg. The reason of this exception has been stated, namely, the great altitude of the Blacksburg station. This fact, for the illustration of which the Blacksburg station was especially selected, indicates that, in platting the latitude curve, some method of reducing it to sea level should be introduced. Just what method is best suited for this purpose can not be stated or even suggested. It is evident, however, that this is a problem which must receive due consideration, and that this calculation must be a special one for each case. For instance, the effect of altitude on temperature on a mountain plateau like that of Blacksburg would be very different from the effect of the same altitude upon the temperature on a vast plain like that extending around Ames, Iowa, or even Lexington, Ky. These problems are of the utmost meteorological and scientific import, and the depression in the curve of latitude, as illustrated in this chart by the Blacksburg station, is of the greatest interest. Eliminating this one point, it will be seen that the content of sugar varies practically with the latitude, the only other exception to this being the data from the Lafayette station, which from the first of the collaborative studies have been found to be very erratic.

The curve showing the percentage of sunshine is broken, because no data for the Madison station is procurable. Kentucky, as will be seen, had the maximum percentage of sunshine platted, namely, 75 per cent. The lowest percentage of sunshine was that of the Blacksburg station, namely, 53.7 per cent. The next highest was that of the stations at Ames and Lafayette, they having the same percentage, namely, 69.9 per cent. The percentage of sunshine seems to have but little effect upon the sugar content, and it appears to be well established that the chemical activities of the sun's light, in promoting the condensation

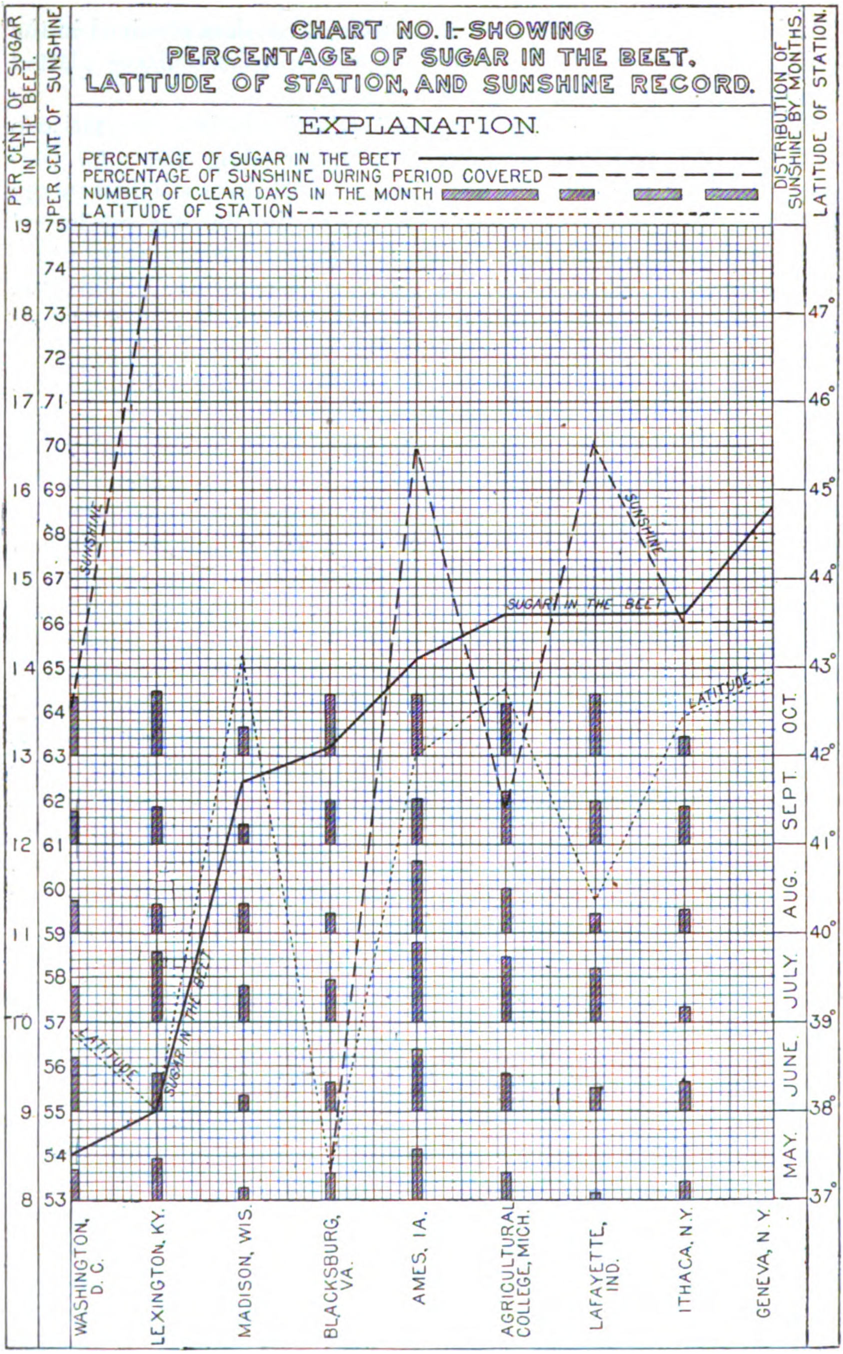

FIG. 1.—Showing percentage of sugar in the beet, latitude of station, and sunshine record.

of carbohydrate molecules in the chlorophyll cells, are not notably diminished by filtration through aqueous vapor.

The distribution of the sunshine as indicated by the number of clear days in each month is also an interesting part of the problem. From the chart, Washington and Ames appear to have had the most uniform distribution of clear days—that is, the number was quite uniform for each month. The smallest number of clear days was experienced at Madison; especially is this true of May, June, and September. The largest number of clear days was experienced at Ames, July and August being conspicuous in this particular. Ithaca stands next to Madison in regard to the small number of clear days, having almost the same record. As in the case of sunshine, it must be said that the actual predominance of clear days is not to be reckoned as an important factor, except in so far as it may indicate drought, and thus interfere with the magnitude of the crop. Thus the large number of clear days at Ames was accomplished by excessively dry weather in 1901.

Chart No. 2 shows the percentage of sugar in the beet, the purity of the juice, the temperature, and the average length of day at the station. The most interesting feature of this chart is the relation between the average length of day and the sugar in the beet. Eliminating, as in the case of latitude, the Blacksburg station, where the days, on account of the southern latitude, were short, and ignoring the slight variation at Lafayette, we find that there is a general agreement in the direction of the two curves, representing the percentage of sugar in the beet and the length of the day. In other words, it may be generally stated, as the result of an inspection of this chart, that the percentage of sugar in the beet increases with the length of the day. This is in harmony with the commonly accepted theory of the correlation of the functional activity of the chlorophyll cells and the light of the sun. Under the same general conditions it is evident that the longer the hours of activity the greater the amount of work accomplished; hence, with longer hours of sunlight the quantity of carbohydrates formed will be greater.

The temperature curve, as in chart 2 of Bulletin 64, crosses the curve of sugar content, but not symmetrically, as in the chart just mentioned. One reason of this lack of symmetry is the low temperature of the Blacksburg station. On the other hand, a phenomenally high temperature is recorded for the Lafayette station. While showing more irregularities than the curves representing latitude and length of day, it is yet evident that the tendency of the sugar is to diminish as the temperature increases. The purity curve, as was to be expected, follows in general the percentage of sugar in the beet.

Chart No. 3 shows the percentage of sugar in the beet, the altitude of the station, and the rainfall record, both the totals and the distribution by months.

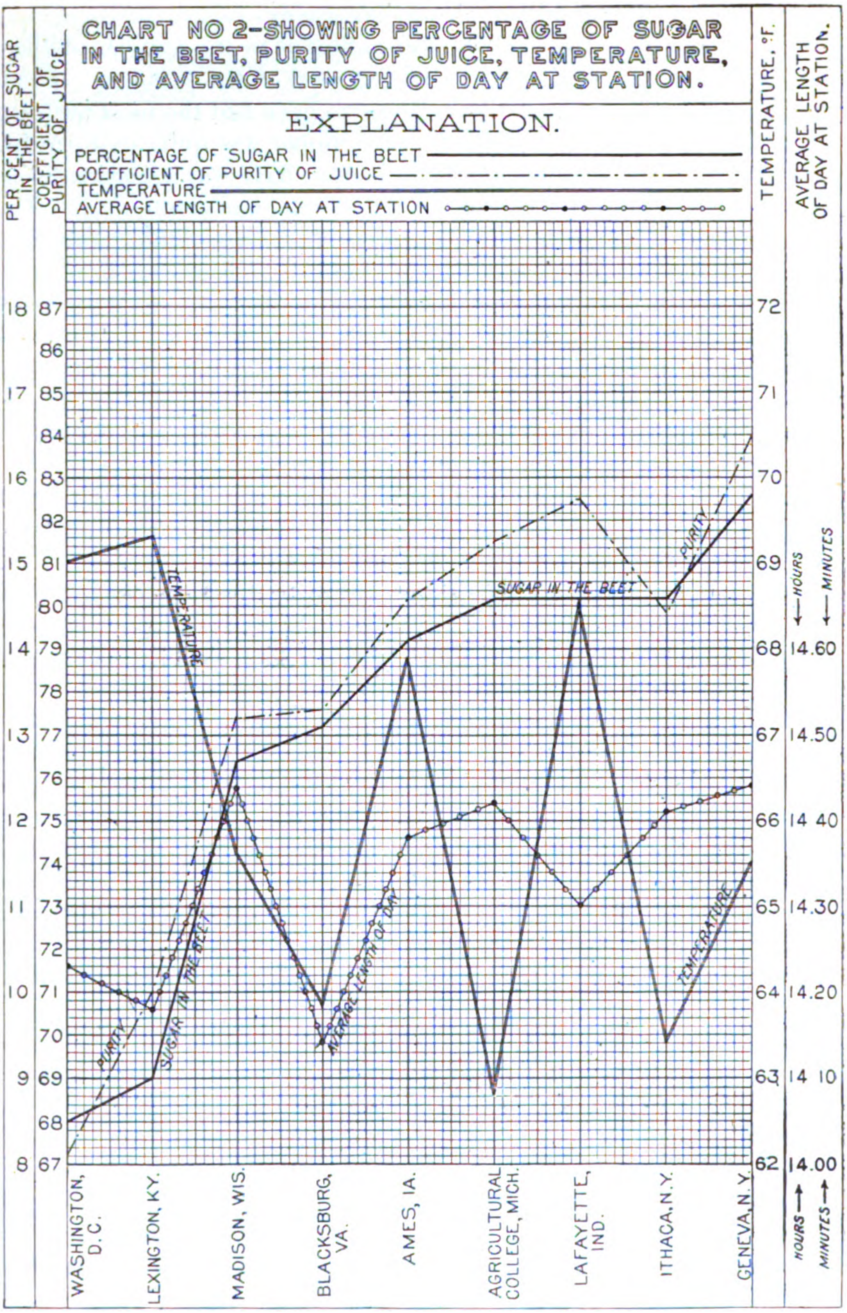

FIG. 2.—Showing percentage of sugar in the beet, purity of juice, temperature, and average length of day at station.

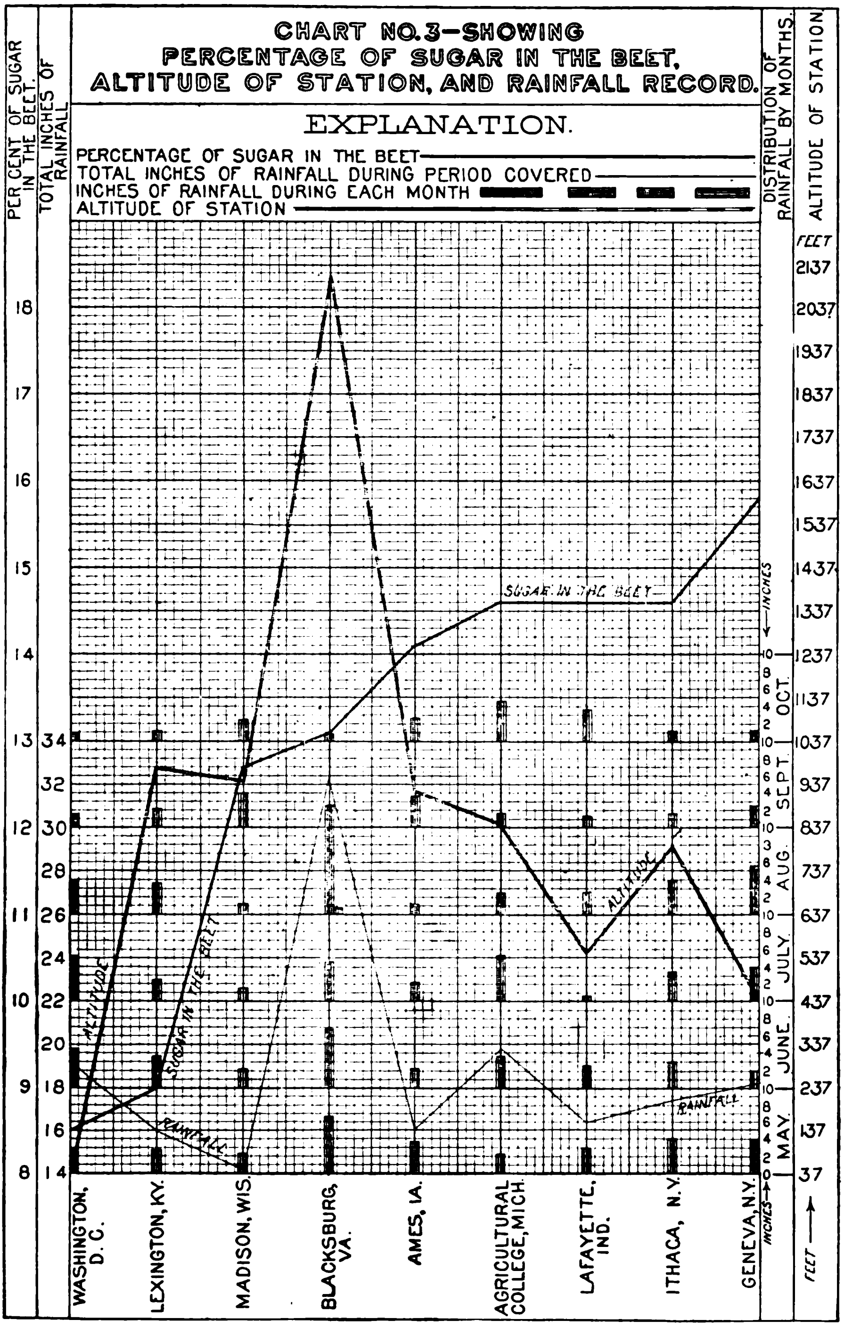

FIG. 3.—Showing percentage of sugar in the beet, altitude of station, and rainfall record.

The remarkable curve showing the altitude of the Blacksburg station is first to be noted. No more striking effect of environment on the composition of the beet could be given than that shown here. It is the altitude in this case which has secured so high a content of sugar in the beet. This station, which has the lowest latitude, shows almost the mean percentage of sugar found at the other stations. Another important effect of altitude is shown at the Washington station, which is practically at sea level, where the lowest altitude coincides with the lowest percentage of sugar. The only proper way to study the effect of altitude, as has already been intimated, is in connection with other physical features of the environment, such as mountain ranges, broad and extensive plateaus, etc. Altitude evidently does not in every case tend to increase the content of sugar. This is illustrated in the case of Ithaca and Geneva. Ithaca is almost 400 feet higher than Geneva and yet the sugar content of the beets grown at Geneva in the two seasons covered by this experiment has been higher than at Ithaca.

The rainfall, as is shown by the curve, was smallest at Madison. Other areas receiving a small rainfall were Ames, Lafayette, and Lexington. The greatest amount of rainfall was at Blacksburg, and the next greatest at Agricultural College, Mich., and at Washington. The actual amount of rainfall does not have so great an influence on the composition of the beet as does its distribution. It has been shown that excellent beets can be produced with a rather deficient supply of rain, as was the case at Madison, at Ames, and at Lafayette. On the other hand, an excessive amount of rain is not necessarily destructive of sugar content, as is illustrated by the plat at the Blacksburg station. In other words, it may be stated that, given a sufficient quantity of water to secure normal growth, the beet is not very sensitive either to a slightly diminished or a slightly increased supply. It is important, however, that the rainfall be not too great in September, which is the period of ripening, nor in October, the season of harvesting.

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U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 75.

H. W. WILEY, CHIEF OF BUREAU.

SUGAR-CANE CULTURE IN THE SOUTHEAST

FOR THE MANUFACTURE OF TABLE SIRUP.

BY

H. W. WILEY,
Chief of the Bureau of Chemistry.

I. FERTILIZER EXPERIMENTS ON SUGAR CANE,
Conducted by W. B. RODDENBERY, *Cairo, Ga.*

II. REPORTS OF SPECIAL AGENTS ON SUGAR-CANE CULTURE,
Introduction by G. L. SPENCER, *Chief of Sugar Laboratory.*

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LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., April 4, 1903.

Sir: I submit herewith for your inspection and approval a manuscript describing the agricultural experiments conducted in Georgia, Alabama, Mississippi, and Florida during the year 1902 in connection with the investigations of the Department of Agriculture relative to the manufacture of table sirup, and recommend its publication as Bulletin No. 75 of this Bureau.

Respectfully,

H. W. Wiley,
Chief of Bureau.

Hon. James Wilson,
Secretary of Agriculture.

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SUGAR-CANE CULTURE IN THE SOUTHEAST

FOR THE MANUFACTURE OF TABLE SIRUP.

I. FERTILIZER EXPERIMENTS ON SUGAR CANE.

(Conducted by W. B. Roddenbery, Cairo, Ga.)

In harmony with an act of Congress approved June 3, 1902, a series of experiments was commenced in various sections of the South to ascertain the methods in use for making table sirup from sorghum, sugar cane, and other sugar-producing plants. The funds for this purpose were not available for use before July 1. Fortunately, through the liberality of Mr. W. B. Roddenbery, of Cairo, Ga., a series of experiments was begun early in the spring which would not have been possible without his assistance.

ORGANIZATION OF THE EXPERIMENTS.

On February 25, 1902, a letter was received from Mr. Roddenbery inclosing a memorandum of proposed agricultural experiments with sugar cane, and asking that such suggestions and modifications as were deemed advisable be made. The plans submitted for approval, which in the main were excellent, are outlined below, followed by the modifications made:

It is proposed that the experiments be made on 24 plats of three rows each, 70 yards long, of ordinary pine land which was planted in collards in the winter of 1900–1901, and in sweet potatoes in the summer of 1901. The land was rooted by hogs during the fall and broken broadcast by a two-horse plow in January, 1902. The cane is to be planted in 4½-foot rows about March 1.

Two one-horse turn-plow furrows are to be thrown out, leaving a small ridge or balk to be burst by the 8-inch round shovel on the guano distributer as the guano[a] is being put in the furrow. The cane is to be planted in the same furrow on top of the guano and covered with two 7-inch round-shovel furrows. The second application of guano will be put out on both sides of each row by a guano distributer when a good stand of cane is up. The third application is to be made in the same manner when the cane is laid by. Subsequent cultivation is to be carried on in the usual way, the cultivations being all alike and made at the same time.

The experiments are to be repeated in every detail on another piece of pine land that was planted in velvet beans in 1901, a very rank and heavy growth having been turned under in December, 1901.

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[a] In this section the term "guano" is applied to any fertilizer containing acid phosphate, nitrogen, and potash.

For the sandy soil of the fields used the following formula for a standard, normal fertilizer is proposed, the standard amount being 1,200 pounds per acre, used in two applications:

	Pounds.
16 per cent acid phosphate	1,200
8 per cent upland meal	400
19 per cent nitrate of soda	200
50 per cent muriate of potash	200

In the application of 1,200 pounds of such a fertilizer to an acre, the land would receive the following amounts of plant food: 720 pounds of acid phosphate, 240 pounds of cotton-seed meal, 120 pounds of nitrate of soda, and 120 pounds of muriate of potash.

In order to ascertain the best manner of applying the above formula and also the quantity per acre that would give the most profitable results the following applications of this mixture were made to plats 1 to 10, inclusive:

TABLE I.—*Experiments with normal fertilizer on plats 1–10.*

Plat No.	Pounds per acre.	Method of application.
1	1,200	Broadcast before planting and turn under.
2	1,200	One application in furrow with cane.
3	1,200	Two applications; one when planted, one in spring.
4	1,200	Three applications; one when planted, one in spring, one in summer.
5	2,000	Broadcast.
6	2,000	One application.
7	2,000	Two applications.
8	2,000	Three applications.
9	800	One application.
10	800	Two applications.

To ascertain the relative value of the ordinary and the lower grade fertilizers generally used under cane, plat No. 11 is to receive 1,200 pounds per acre in two applications of the following formula:

	Pounds.
13 per cent acid phosphate	1,000
8 per cent cotton-seed meal	600
Kainit	400

To ascertain the relative importance of each of the plant foods, and as a guide to the most desirable proportions to be used, the following experiments are to be made on plats Nos. 12 to 17, inclusive, in which each ingredient is omitted and doubled in turn. Two applications of the fertilizer will be made:

TABLE II.—*Experiments with varying proportions of plant foods on plats 12–17.*

Plat No.	Pounds per acre.			
	Acid phosphate.	Cotton-seed meal.	Nitrate of soda.	Muriate of potash.
12	720	240	120	None.
13	720	None.	None.	120
14	None.	240	120	120
15	1,440	240	120	120
16	720	480	240	120
17	720	240	120	240

As an experiment in the relative value of cotton-seed meal and nitrate of soda ammoniates the following compounds are to be applied to plats Nos. 18 and 19. Two applications were made:

TABLE III.—*Cotton-seed meal and nitrate of soda compared.*

Plat No.	Pounds per acre.			
	Acid phosphate.	Cotton-seed meal.	Nitrate of soda.	Muriate of potash.
18	675	525	....	110
19	756	....	221	126

As an additional experiment as to the relative value of cotton seed as an ammoniate (cost, 25 cents per bushel) and cotton-seed meal (at $23 per ton), when applied in the usual quantity used by the best cane growers, i. e., 50 bushels to the acre, the following mixtures were tried on plats Nos. 20 and 21, two applications being made as usual:

TABLE IV.—*Comparison of cotton seed and cotton-seed meal.*

Plat No.	Pounds per acre.		
	Acid phosphate.	Cotton seed.	Muriate of potash.
20	720	[a] 728	120
21	650	[b] 1,720	97

[a] Equivalent to 24 bushels, which at 25 cents per bushel cost $6.

[b] Equivalent to 50 bushels, or 525 pounds of meal, which at $23 per ton cost $6.03. This is about the amount used by the smaller cane planters who do not count the cost of seed.

To ascertain the relative value of muriate of potash and kainit, the following fertilizers were applied to plats Nos. 22 and 23. Two applications were made:

TABLE V.—*Kainit and muriate of potash compared.*

Plat No.	Pounds per acre.				
	Acid phosphate.	Cotton-seed meal.	Nitrate of soda.	Kainit.	Muriate of potash.
22	720	240	120	480	....
23	720	240	120	....	120

Plat No. 24 received no fertilizer, being used as a check plat. The cane from each plat is to be accurately weighed and the juice tested by the Baumé saccharimeter.

The following suggestions were made to Mr. Roddenbery in modification of the plans submitted and were put into effect:

Instead of having only three rows in each plat 4 feet apart, four were planted, inasmuch as the outside rows in one plat might be slightly affected by the fertilizer applied to the next one. The space of one row was allowed between each plat, either planted with cane of which no account was taken or left fallow. With each series of experiments a plat was introduced containing no fertilizer. Arrangements were also made for special analysis of the fertilizer used, franked packages for sending samples of them being provided and directions given for securing a fair sample. These suggestions were adopted by Mr. Roddenbery, and later arrangements were made, after the money appropriated by Congress became available, to send a special agent to Cairo for the purpose of assisting in the harvesting and weighing of the crops and in securing samples for analysis.

AGRICULTURAL DATA.

The two fields finally selected for this experiment are designated as A and B. The soil is a sandy loam, with clay subsoil and of low fertility, being fairly representative of the soils of that section. In the growth of all standard crops in this locality where large and profitable yields are desired, fertilizers are universally applied. The water supply is usually sufficient and the water comes near the surface. The season, taken as a whole, was a favorable one and the tonnage probably better than the average. Each field was 70 yards wide and 140 yards long, and was divided into 24 plats, each consisting of four rows 4 feet apart. The fertilizer was applied only to the three rows, the fourth row being unfertilized as a check between the plats.

Field A is situated in the middle of a 15-acre field of sugar cane and was thus subjected to the ordinary conditions of growth. This field has been in cultivation about twenty years and is in a low state of fertility, and most of the available plant food was supplied by the fertilizer. The field was planted to sweet potatoes in 1901, was plowed with a two-horse disk plow in January, 1902, and again plowed just before the cane was planted. On April 3, Field A was planted with stubble cane cut short and placed end to end in the row.

Field B is situated in the middle of a 25-acre field of sugar cane one-half mile distant from Field A. The soil is similar in every respect to Field A, but has been in cultivation only five or six years and is in a much higher state of fertility than Field A. Field B was planted to velvet beans in 1901 and a very rank growth of these beans was plowed under in the autumn of 1901, the field being again plowed just before the cane was planted, namely, on April 4. Stubble cane was also used for this field, which was planted in the same manner as Field A. No special cultivation was given the experimental plats, but they received exactly the same attention as the whole field in which they were situated.

Mr. Ralph Hoagland, of Minnesota, was appointed a special agent of the Bureau of Chemistry and detailed to assist Mr. Roddenbery in the harvesting and testing of the crop.

The agricultural data were obtained directly at the fields and factory by weighing a part of the crop and calculating the entire yield from the area harvested. Samples from each plat were separately milled and samples of juices from the mill were secured and transmitted to the Washington laboratory for analysis. The plats on Field A and Field B were treated precisely alike in every particular.

The following observations as to stand were made on the two fields on the dates given:

TABLE VI.—*Stand on Fields A and B.*

Plat No.	April 28.		May 23.		Plat No.	April 28.		May 23.	
	A.	B.	A.	B.		A.	B.	A.	B.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	80	50	85	85	13	40	40	85	85
2	20	15	90	80	14	50	50	85	80
3	40	40	95	90	15	40	75	85	90
4	40	50	95	90	16	20	10	85	60
5	80	90	100	95	17	25	15	90	60
6	10	10	40	50	18	50	20	90	75
7	15	25	80	70	19	25	60	85	85
8	25	25	85	65	20	80	50	100	70
9	25	35	85	75	21	75	40	100	60
10	20	20	80	75	22	15	10	80	65
11	15	15	80	60	23	60	30	85	80
12	25	40	85	75	24	50	40	75	50

The percentages expressing condition of the crop as given in the following table are used in the same sense as such numbers are used by the Bureau of Statistics of this Department in its crop reports:

TABLE VII.—*Condition and height of cane, May 23–October 13.*

Plat No.	Condition, May 23.		Height,[a] July 10.		Height,[a] July 30.		Height,[b] Oct. 13.	
	A.	B.	A.	B.	A.	B.	A.	B.
	Per cent.	*Per cent.*	*Inches.*	*Inches.*	*Inches.*	*Inches.*	*Inches.*	*Inches.*
1	90	80	54	49	75	68	60	60
2	80	75	54	50	75	71	62	49
3	95	80	53	50	74	73	61	60
4	90	85	52	49	73	72	63	60
5	100	90	58	52	82	75	64	68
6	50	50	52	42	72	64	62	64
7	90	70	54	51	78	74	63.5	68
8	90	65	55	47	76	68	66	64
9	90	75	53	48	72	71	56	58
10	85	80	52	49	72	69	51	58
11	85	70	53	46	72	66	58	54
12	85	75	54	47	69	67	52	58
13	85	85	50	49	68	67	53	60
14	80	75	46	42	63	60	57	68
15	90	90	54	50	73	72	60	68
16	85	65	55	44	75	69	61	66
17	90	70	54	47	74	70	56	64
18	90	75	54	45	75	65	60	62
19	85	90	49	49	73	71	58	66
20	95	90	51	47	68	63	53	60
21	100	70	55	45	79	65	61	64
22	75	70	51	44	76	64	59	67
23	90	75	58	49	79	68	59	67
24	70	65	44	85	59	48	46	36

[a] Height measured to top of leaves.
[b] Height measured to top of first red joint.

The general condition of Field A on October 15 was not as good as that of Field B. In Field A the dead leaves were about halfway up the stalks. The double phosphoric acid plat (No. 15) had the largest, tallest cane and the unfertilized plat (No. 24) the smallest. The cane in Field B did not show as many dead leaves, but the plats of the two fields appeared about the same, except that the "no ammonia" plat (No. 13), Field B, had very good cane while the same plat in Field A had poor cane.

The general progress of the cane in the two fields between October 17 and November 3 is shown in the following tabulated observations:

TABLE VIII.—*General condition of Field A, October 17, 1902.*

Plat No.	No. of stalks.[a]	General condition of cane.
1	375	Fair quality of cane.
2	428	Trifle better than 1.
3	437	About the same as 2.
4	408	Do.
5	462	Good cane; better than 3 and 4.
6	359	Light stand, fair cane, too much fertilizer in drill.
7	451	About the same as 5.
8	412	Do.
9	326	Much poorer than 8, light stand.
10	324	Same as 9.
11	328	About the same as 10.
12	320	Rather poor cane.
13	321	Very poor cane, not healthy green.
14	343	Fair cane, far better than 13.
15	411	Heavy, fine cane.
16	469	Do.
17	408	Very fair cane, not as good as 15.
18	415	Not as good as 17; good cane.
19	328	Poor stand, fair quality cane.
20	350	Lighter cane than 19, but not so many gaps.
21	416	Good cane, much better than 20.
22	398	Good cane, but not as good as 21.
23	439	Very good cane.
24	255	Very poor, short cane.

[a] Number of stalks of cane of fair size in middle row of each plat.

OBSERVATIONS ON FIELD A, NOVEMBER 3.

This field of cane will yield fewer tons of cane per acre than Field B, but at this time is much nearer maturity, the dead leaves reaching nearly up to the tuft at the top of the cane.

Plat 1. Fair cane, good stand; not as good as Field B, plat No. 1.
Plat 2. Fairly good cane, stand better than plat No. 1.
Plat 3. About the same as plat No. 2.
Plat 4. About the same as plats Nos. 2 and 3.
Plat 5. Very good cane, not as good as Field B, plats Nos. 3 and 5.
Plat 6. Good cane, but not as good as plat No. 5.
Plat 7. Very good cane, better than plat No. 6.
Plat 8. About the same as plat No. 7.

Plat 9. Fair cane only, fair stand, shows need of more fertilizer.
Plat 10. Same as plat No. 9.
Plat 11. Fairly good cane, better than plats Nos. 9 or 10.
Plat 12. Cane poor to fair, poor stand.
Plat 13. Very poor cane, poorer than plat No. 12; short and yellow.
Plat 14. Fairly good cane, better than plats Nos. 12 and 13.
Plat 15. Very good cane, better than plat No. 14; a trifle better than plats Nos. 3 and 4.
Plat 16. Very good cane, better than plat No. 15; greater diameter of stalk, yield will be a trifle heavier.
Plat 17. Good cane, not quite as heavy as plat No. 16.
Plat 18. Good cane, about the same as plat No. 17.
Plat 19. Good cane, about the same as plat No. 18.
Plat 20. Very poor cane, but little better than the unfertilized plat.
Plat 21. Good cane, about the same as plats Nos. 3 or 4.
Plat 22. Very good cane, better than plat No. 21.
Plat 23. Very good cane, better than plat No. 22.
Plat 24. Very poor cane; short, yellow.

TABLE IX.—*General condition of Field B, October 20, 1902.*

Plat No.	No. of stalks.[a]	General condition of cane.
1	454	Heavy cane, much better than Field A, plat No. 1.
2	478	Very heavy cane, about the same as plat No. 1.
3	496	Very heavy cane, trifle heavier than plat No. 2.
4	456	Very heavy cane, about the same as plat No. 3.
5	542	Very heavy cane, heavier than plat No. 4; badly twisted.
6	451	Good cane, only fair stand, stalks smaller than plat No. 5.
7	481	Heavy cane, but stalks smaller than plat No. 4.
8	461	Rather heavy, larger stalks than plat No. 7.
9	437	Not as good stand nor cane as plats Nos. 7 and 8.
10	414	Cane good, about the same as plat No. 9.
11	398	Fair cane, about the same as plat No. 10.
12	389	Only fair cane, about the same as plat No. 11.
13	397	Good cane, much better than plat No. 12.
14	340	Very poor cane, short; light stand, but little better than plat No. 24.
15	493	Very heavy stand, tall cane, fair diameter; too thick, too much phosphoric acid.
16	392	Good cane, large diameter, fair height; not as good as plat No. 15.
17	377	Good cane, about the same as plat No. 16.
18	423	Very good cane, nearly as good as plat No. 15.
19	436	Do.
20	390	Good cane, fair stand.
21	335	Good cane, poor stand.
22	377	Good cane, stand uneven.
23	360	Good cane, not as good as plats Nos. 1 and 2.
24	262	Poor stand of cane, a trifle better than plat No. 24, Field A.

[a] Number of stalks of fair-sized cane in middle row of each plat.

OBSERVATIONS ON FIELD B, OCTOBER 30, 1902.

The dead leaves were hardly halfway up on the cane.
Plat 1. Very heavy cane, good stand.
Plat 2. Perhaps trifle heavier than plat No. 1.

Plat 3. About the same as plat No. 2.

Plat 4. About the same as plat No. 2.

Plat 5. Very heavy cane, perhaps a trifle heavier than plat No. 4; badly twisted and down, worse in that respect than plat No. 4.

Plat 6. Heavy cane, but rather poor stand; shows injurious effect of large amount of guano in drills.

Plat 7. Extra heavy cane, heavier than plats Nos. 3 and 4; large stalks, rather badly twisted.

Plat 8. About the same as plat No. 7.

Plat 9. Very good cane, about the same as plat No. 1.

Plat 10. Same as plat No. 9.

Plat 11. Good cane, fair stand, cane perhaps trifle better than plat No. 10.

Plat 12. Only fair cane, poor stand, not as good as plats Nos. 9 and 10; shows need of some fertilizing element.

Plat 13. Good cane, much better than plat No. 12; stalks of good size; shows need of but little ammonia.

Plat 14. Fair cane only, not as good as plat No. 13, about the same as plat No. 12; shows need of another fertilizing element.

Plat 15. Very heavy stand, tall cane, stalks of small diameter and joints long, due to rapid growth(?). Yield will probably not be greater than that of plats Nos. 5 and 7.

Plat 16. Very heavy cane, large diameter, good height but not as tall as plat No. 15, fair stand.

Plat 17. Very good cane and stand; diameter yield will be better than plat No. 16 and nearly as good as plat No. 15.

Plat 18. Very good cane, about the same as plats Nos. 3 and 4.

Plat 19. Seems to be a trifle better, heavier cane than on plat No. 18.

Plat 20. Cane fair to good; fair stand.

Plat 21. Very good cane but poor stand, cane about as plats Nos. 3 and 4; yield hardly as large.

Plat 22. Very good cane, fair stand; about the same as plats Nos. 3 and 4.

Plat 23. Very good cane, about the same as plats Nos. 1 and 22; no better than plot No. 22. Can see no advantage in mixing guano with soil before planting.

Plat 24. Poor cane, poor stand, but better than plat No. 24, Field A.

COMPOSITION AND COST OF FERTILIZERS.

The composition of the samples of fertilizers received from Mr. Roddenbery were analyzed in this Bureau with the following results:

TABLE X.—*Analyses of fertilizers.*

Serial No.	Variety.	Phosphoric acid.				Potash. (K_2O.)	Nitrogen. (N.)	Ammonia. (NH_3.)
		Water soluble.	Citrate soluble.	Available.	Total.			
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
23991	Acid phosphate	10.27	3.09	13.36	15.30	....	0.13	0.16
23992	Acid phosphate[a]	15.14	2.82	17.96	18.16	....	.17	.21
23993	Cotton-seed meal	.63	1.32	1.95	2.53	1.82	6.54	7.94
23994	Nitrate of soda	....	....	....	....	1.52	13.93	16.91
23995	Kainit	....	....	....	....	12.49	.02	....
23996	Muriate of potash	....	....	....	....	50.72	.03	....
	Normal[b]	....	....	11.95	....	5.58	....	3.53

[a] Used in composition of normal fertilizer.

[b] Calculated.

In the following table is given the cost per ton of the different fertilizers mentioned in Table X and the cost per pound of the principal fertilizing elements contained therein. In Table XII will be found the cost of the various mixtures as applied, per acre.

TABLE XI.—*Cost of fertilizers.*

Fertilizer.	Cost per ton.	Amount of principal fertilizing elements per ton.		Cost of principal fertilizing elements per pound.
		Kind.	Quantity.	
	Dollars.		*Pounds.*	*Cents.*
Acid phosphate (16 per cent)	12.50	Phosphoric acid	359	3.5
Acid phosphate (13 per cent)	11.80	.....do	267	4
Sodium nitrate	48.00	Nitrogen	338	14.2
Muriate of potash	45.00	Potash	1,014	4.4
Cotton-seed meal	23.50	Nitrogen	158.8	14.8
Cotton seed	16.66	.....do	76	21
Kainit	13.50	Potash	258.8	5.2

In the above table it will be noted that in estimating the cost of the fertilizing elements per pound only the principal one has been selected and the whole cost of the fertilizer charged to that. This is of course an unobjectionable method, provided the fertilizer contains only one element, as was the case with the acid phosphates of kainit and muriate of potash above mentioned. In the case of cotton-seed meal the phosphoric acid and the potash, however, are worth something, and hence the value of these should be deducted from the total expense, and their value should not be charged to the nitrogenous element alone. In making this change we find that the cotton-seed meal contained 1.82 per cent of potash, or 36.4 pounds per ton, which, at 5 cents per pound, amounts to $1.82. The cotton-seed meal contained also 1.96 per cent of available phosphoric acid, which, at 4 cents per pound and 39.2 pounds per ton, amounts to $1.57. Potash and phosphoric acid together, therefore, are worth $3.39. Deducting this from the price per ton of cotton-seed meal, namely, $23.50, we find the value of the nitrogenous element to be $20.11. Dividing this by the number of pounds of nitrogen reckoned as ammonia, namely, 158.8, we find the price of the ammonia to be 12.6 cents per pound.

In determining the cost of phosphoric acid and potash per unit it will be seen that the high-grade fertilizers furnish these elements at a cheaper rate than the low grade. For instance, the guaranteed 16 per cent acid phosphate supplies phosphoric acid at 3½ cents a pound, while the guaranteed 13 per cent available phosphoric acid supplies this element at 4 cents per pound. It will be noticed in both cases that the acid phosphates were above the guarantee in available phosphoric acid, one being 0.36 per cent and the other 1.96 per cent above the guarantee.

COMPARISON OF AGRICULTURAL AND CHEMICAL DATA.

A summary of the agricultural and chemical data obtained in the fertilizer experiments are given in Table XII. The analytical results in this table, as well as the figures given on the cane juices throughout the reports of the special agents in Part II, were obtained by the assistants in the sugar laboratory of this Bureau working under the direction of Mr. G. L. Spencer, the sucrose and purities having been determined by A. Wilbur Bache and the reducing sugars by H. W. Houghton.

The practical result of the experiment that would naturally be considered first is the tonnage. This, however, is not an absolute standard, as a study of the chemical data in connection with the tonnage will show, the former being of the greatest importance in sirup making in so far as quality is concerned. A glance at the analytical data will show that the juices coming from the cane of Field A were very distinctly superior, both in sugar content and in purity, to the juices obtained from the cane of Field B. It is evident, therefore, that a higher grade of sirup would be made from the cane grown on Field A.

TABLE XII.—*Summary of agricultural and chemical data on fertilizer experiments, Fields A and B.*

No. of plat.	Sucrose.		Reducing sugar.		Purity.		Yield per acre.		Number of stalks.		Cost of fertilizer per acre.	Fertilizers: Composition,[a] quantity, and method of application.[b]
	A.	B.	A.	B.	A.	B.	A.	B.	A.	B.		
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*			*Tons.*	*Tons.*				
1........	16.22	14.97	.40	.80	91.52	89.05	19.96	26.88	1,227	1,340	$13.50	1,200 pounds normal fertilizer to acre; broadcast before planting and turned under.
2........	15.51	15.10	.51	.89	90.12	88.82	20.56	27.64	1,231	1,410	13.50	1,200 pounds normal fertilizer to acre; 1 application in furrow with cane.
3........	15.66	15.56	.44	.71	91.04	88.89	20.36	28.72	1,254	1,423	13.50	1,200 pounds normal fertilizer to acre; 2 applications when planted, May 23.
4........	15.76	13.90	.39	1.18	90.58	86.34	19.60	28.00	1,179	1,381	13.50	1,200 pounds normal fertilizer to acre; 3 applications when planted, May 23—July 10.
Average.	15.79	14.88	.44	.90	90.82	88.28	20.12	27.81	1,225	1,389	13.50	
5........	16.04	13.36	.75	1.18	92.18	84.56	23.24	35.36	1,367	1,574	22.50	2,000 pounds normal fertilizer to acre; broadcast and plowed under before planting.
6........	14.55	12.68	.56	1.48	87.88	81.81	19.40	23.84	1,091	1,323	22.50	2,000 pounds normal fertilizer to acre; 1 application in furrow with cane.
7........	15.21	14.25	.52	.98	91.06	86.88	24.60	39.40	1,475	1,431	22.50	2,000 pounds normal fertilizer to acre; 2 applications May 23—July 10.
8........	15.18	13.34	.54	1.25	89.82	88.86	24.60	28.88	1,307	1,322	22.50	2,000 pounds normal fertilizer to acre; 3 applications when planted, May 23—July 10.
Average.	15.25	13.41	.59	1.22	90.24	85.54	22.96	31.87	1,310	1,413	22.50	
9........	15.03	14.09	.53	1.04	90.00	86.42	16.88	25.76	1,102	1,126	9.00	800 pounds normal fertilizer to acre; 1 application in furrow with cane.
10........	15.58	14.68	.44	.92	88.02	86.86	21.48	28.82	1,096	1,389	9.00	800 pounds normal fertilizer to acre; 2 applications.
Average.	15.35	14.39	.49	.98	89.01	86.64	19.18	27.04	1,099	1,258	9.00	
12........	15.27	14.74	.55	.91	89.83	88.26	13.56	19.76	1,036	1,150	10.20	720 pounds 16 per cent acid phosphate; 240 pounds cotton-seed meal; 120 pounds nitrate of soda; *no potash.*
13........	16.41	14.52	.37	.92	....	87.47	10.80	24.40	914	1,190	7.20	720 pounds 16 per cent acid phosphate; *no ammonia;* 120 pounds muriate of potash.
14........	16.25	14.79	.33	1.18	91.52	84.28	14.72	18.80	1,027	1,139	8.40	*No acid phosphate;* 240 pounds cotton-seed meal; 120 pounds nitrate of soda; 120 pounds muriate of potash.
Average.	15.98	14.68	.42	1.00	90.68	86.67	13.03	20.99	992	1,160	8.60	
15........	16.01	14.01	.46	.91	91.50	86.48	18.24	31.50	1,223	1,493	17.40	*1,440 pounds acid phosphate (double);* 240 pounds cotton-seed meal; 120 sodium nitrate; 120 pounds muriate of potash.
16........	14.93	13.01	.64	1.52	88.86	82.28	23.44	24.50	1,406	1,235	18.60	720 pounds acid phosphate; *480 pounds cotton-seed meal (double); 240 pounds sodium nitrate (double);* 120 pounds nitrate of potash.
17........	16.31	12.66	.39	1.29	91.12	80.13	19.84	25.60	1,255	1,184	15.60	720 pounds acid phosphate; 240 pounds cotton-seed meal; 120 pounds sodium nitrate; *240 pounds muriate of potash (double).*
Average.	15.75	13.23	.50	1.24	90.49	82.96	20.51	27.20	1,295	1,304	17.20	

[a] When cotton seed was used, instead of cotton-seed meal, it had the following composition: Available phosphoric acid, 1.27 per cent; nitrogen as ammonia, 3.8 per cent potash, 1.17 per cent.

[b] Unless otherwise stated two applications of the fertilizer were made.

TABLE XII.—*Summary of agricultural and chemical data on fertilizer experiments, Fields A and B*—Continued.

No. of plat.	Sucrose.		Reducing sugar.		Purity.		Yield per acre.		Number of stalks.		Cost of fertilizer per acre.	Fertilizers: Composition, quantity, and method of application.
	A.	B.	A.	B.	A.	B.	A.	B.	A.	B.		
TO ASCERTAIN RELATIVE VALUE OF ORIDINARY AND LOW-GRADE FERTILIZERS GENERALLY USED WITH CANE.												
11........	*Per ct.* 15.63	*Per ct.* 14.23	*Per ct.* .40	*Per ct.* 1.00	90.33	87.30	*Tons.* 15.04	*Tons.* 24.08	997	1,248	9.36	1,200 pounds to acre (1,000 pounds 13 per cent acid phosphate; 600 pounds cotton-seed meal; 400 pounds kainit). Two applications; one when planted, one May 23.
TO ASCERTAIN RELATIVE VALUE OF COTTON-SEED MEAL AND NITRATE OF SODA AS AMMONIATES.												
18........	16.22	15.05	.40	.80	91.64	88.52	17.32	29.00	1,156	1,303	12.90	675 pounds acid phosphate; 525 pounds cotton-seed meal; 110 pounds muriate of potash.
19........	15.56	14.47	.49	.91	91.00	86.10	20.36	29.08	1,212	1,323	12.86	756 pounds acid phosphate; 221 pounds nitrate of soda; 126 pounds muriate of potash.
Average.	15.89	14.76	.45	.86	91.32	87.31	18.84	29.04	1,184	1,313	12.88	
TO ASCERTAIN RELATIVE VALUE OF COTTON SEED AND MEAL.												
20........	16.22	14.52	.42	.86	91.16	86.89	13.80	26.32	1,073	1,227	13.20	720 pounds acid phosphate; 728 pounds cotton seed; 120 pounds muriate of potash.
21........	16.28	14.55	.39	.83	92.50	87.64	19.08	31.62	1,239	1,136	20.62	650 pounds acid phosphate; 1,720 pounds cotton seed; 97 pounds muriate of potash.
Average.	16.25	14.54	.41	.85	91.83	87.27	16.44	28.97	1,156	1,182	16.41	
TO ASCERTAIN RELATIVE VALUE OF KAINIT AND MURIATE OF POTASH.												
22........	16.12	13.26	.44	1.25	91.07	84.43	19.00	26.60	1,123	1,326	13.17	720 pounds acid phosphate; 240 pounds cotton-seed meal; 120 pounds nitrate of soda; 480 pounds kainit.[a]
23........	16.20	14.28	.44	.94	91.53	86.02	19.32	26.60	1,178	1,192	13.50	720 pounds acid phosphate; 240 pounds cotton-seed meal; 120 pounds nitrate of soda; 120 pounds muriate of potash.[b]
Average.	16.16	13.77	.44	1.05	91.30	85.23	19.16	26.60	1,151	1,259	13.34	
24........	15.72	14.17	.44	1.21	91.40	85.33	9.12	12.00	743	841	....	No fertilizer.

[a] Compare also with plat No. 3.

[b] Fertilizer mixed with dirt before planting cane.

This better quality of sirup would naturally command a better price in the market; therefore the actual financial difference in the results between the two fields would not be so great as would appear from a study of the tonnage data alone. In comparing the yields from Fields A and B it must not be forgotten that the actual fertility of Field B is greater than Field A, because it has been a shorter time under cultivation, and in addition to this the crop in Field B was preceded by a leguminous crop, a nitrogen gatherer which was plowed under, thus adding a large amount of available nitrogen to the succeeding crop. In Field A, on the contrary, sweet potatoes had been grown, and this crop adds nothing to the fertility of the soil, but rather tends to diminish it.

The basic data for comparison are found in Plat 24, to which no fertilizer was applied. The yield of cane per ton on this plat was 9.12 tons in Field A and 12 tons in Field B. It is to be regretted that one or two additional check lots were not provided, since the difference in yield in the two plats 24 in Fields A and B is not so great as one would expect. In other words, although Fields A and B were treated exactly alike, if you should add only 3 tons per acre to the yield of Field A you would not reach the yield of Field B in many instances. This fact leads to the inevitable conclusion either that the two plats 24 in each field do not afford a sufficient comparison, or else the previous treatment of Field B made it unusually well adapted to utilize to the very best advantage the plant foods added in the fertilizer. In another experiment there should be at least four unfertilized plats in each field.

Without going over the data in detail it will be interesting to compare the yields per acre on the plats which were treated with the same amount of fertilizer, but applied differently, namely, 1, 2, 3, and 4; 5, 6, 7, and 8; and 9 and 10. It will be evident at once that the normal fertilizer suggested is the best combination of fertilizing material with which experiments were made. The yields per acre, the percentage of sugar, and the purity of the juice are the data on which our judgment must be based.

On Field A with 1,200 pounds of normal fertilizer the average yield was 20.12 tons per acre on plats Nos. 1 to 4, inclusive. On plats Nos. 5 to 8, inclusive, with 2,000 pounds of the normal fertilizer we see that the average yield was 22.96 tons per acre, while on plat No. 24, to which no fertilizer was applied, the yield was 9.12 tons per acre. These data show that on Field A the application of the normal fertilizer more than doubled the yield when applied at the rate of 1,200 pounds; while with the use of 2,000 pounds per acre the yield was increased almost exactly 152 per cent. Where the application dropped to 800 pounds per acre the average yield was also very satisfactory, being almost exactly double the yield without fertilizer. The application of the fertilizer on plat No. 11 was less efficacious, while the fertilizers on plats Nos. 12, 13, and 14 were still less remunerative. Plats Nos. 15, 16, and 17, however, gave good results; plats Nos. 18 and 19 fair results; plat No. 20 poor results; and plats Nos. 21, 22, and 23 also good.

Plats Nos. 12, 13, and 14 perhaps are the most instructive in Field A, for they show that a complete fertilizer is absolutely necessary to give the best results. To plat No. 12 no potash was added, to plat No. 13 no nitrogen, and to plat No. 14 no acid phosphate. The most striking feature of this part of the experiment is that nitrogen is shown to be the most deficient element in the soil of Field A, since when no nitrogen was added the yield per acre was only a little above 1 ton more than when no fertilizer was added. The next most important plant food to the crop is potash, and the third phosphoric acid.

Turning to Field B, we find, of course, a different condition of affairs, for in that field a natural fertility of the soil exists. On plats Nos. 12, 13, and 14 good crops were grown, showing that no fertilizing element was altogether absent. In the blank plat the yield was about 33 per cent higher than in Field A. The fertilizers, as a rule, give larger results on Field B than on Field A. For instance, 1,200 pounds on plats Nos. 1 to 4, inclusive, Field B, produce an average yield of 27.81 tons per acre—that is, an increased average yield of 15.81 tons per acre—while on plats Nos. 5 to 8, 2,000 pounds of normal fertilizer produce an average yield of 31.87—that is, an increased yield of 19.87 tons per acre. In Field B it is also seen that the 800 extra pounds of normal fertilizer applied to plats Nos. 5 to 8, inclusive, produced a yield of nearly 4 tons per acre in excess of the yield of plats Nos. 1 to 4 with 1,200 pounds of fertilizer. If we value the cane at $3 per ton, the 800 pounds of fertilizer produced an increase of crop worth $12 per acre.

In addition to the fertilizers mentioned, barnyard manure is very much prized by cane growers, and it gives much better results than would be expected, judging from its chemical analysis. This is doubtless due to the mechanical changes which the organic matter produces in the soil and to the introduction of the organisms causing fermentation and decomposition, which quickly form considerable quantities of humus, a constituent in which the sandy soil is particularly deficient. The growth of velvet beans, which are either plowed under or used as a pasture by stock, is also found in many instances to give beneficial effects. The velvet bean being a leguminous crop, it would naturally become a nitrogen gatherer, and when the crop is plowed under in the course of time it also tends to add a considerable quantity of humus to the soil.

It is evident that in so far as the artificial fertilization of ordinary sandy soils, of which Fields A and B are types, is concerned, the mixture characterized as normal fertilizer, and consisting of acid phosphate, cotton-seed meal, nitrate of soda, and muriate of potash, affords the best financial returns.

In regard to the quantities to be used, the results of the experiments show conclusively that from 800 to 1,200 pounds per acre will yield the best financial results. While a larger crop is secured on the poorer soils by the use of 2,000 pounds of this mixture to the acre, the increase does not justify the additional cost of the fertilizing materials.

Perhaps the most striking result based upon the data obtained from the plats of the two fields is shown in the fact that the large quantity of nitrogen made available by plowing under the crop of velvet beans on Field B can not be fully assimilated by the growing crop unless the other essential plant foods are supplied. As has been mentioned before, in the unfertilized plat of Field B the yield of cane was a little less than 3 tons greater than on the unfertilized plat of Field A. If, however, the other essential elements of plant food are supplied the increased yield due to the presence of the decaying velvet beans is at once apparent. On plat No. 12 of Field B we find that 720 pounds of acid phosphate were used and a yield of 19.76 tons were secured, which is 7.76 tons above the yield of the unfertilized plat No. 24. In plat No. 13, where 720 pounds of acid phosphate and 120 pounds of muriate of potash per acre were employed, the yield is 24.4 tons, being 12.4 tons in excess of the yield of plat No. 24. On plat No. 14, where 120 pounds of muriate of potash were used, and no acid phosphate, the yield was 18.8 tons, being 6.8 tons above the yield of plat No. 24. On plats Nos. 12 and 14 a considerable quantity of nitrogen was used in the fertilizer, while in plat No. 13 no additional nitrogen was used. The data from these three plats show very conclusively that the velvet beans supplied practically all the nitrogen necessary to produce a large crop. Nevertheless, on comparing plats Nos. 12 to 14, inclusive, with plats Nos. 1 to 4, inclusive, it is seen that where all the essential plant foods are present the addition of an increased amount of nitrogen shows a considerable increase in the yield.

In plats Nos. 18 and 19 a comparison is made of the utility of nitrogen in the form of cotton-seed meal and in the form of nitrate of soda; the quantity of phosphoric acid and potash applied to these two plats were as nearly the same as possible—that is, the phosphoric acid and the potash in the cotton-seed meal added to that given as acid phosphate and muriate of potash would make the quantities of these two fertilizing elements in plat No. 18 the same as those added to the fertilizer applied to plat No. 19. The results of the experiments show that there is practically no difference in the availability of the nitrogen applied as cotton-seed meal and as nitrate of soda, the tonnage in the plats compared being almost identical in Field B. In plats Nos. 20 and 21 a comparison is made between the nitrogen in cotton-seed meal and in cotton seed. The quantities of nitrogen, phosphoric acid, and potash applied on the two plats were practically the same. In this case it appears that nitrogen in the form of cotton seed was more effective than in the form of cotton-seed meal. Additional data, however, would be required before asserting positively that such is the case. In plats Nos. 22 and 23 a comparison is made between the value of potash as kainit and potash as muriate. The data obtained show that there is no difference in the availability of the potash in these two forms. In plat No. 11 a fertilizer was employed in which the phosphoric acid and potash was supplied from low-grade materials instead of the high grade as used in preparing the normal fertilizer. The result of this one test is not definite, but seems to indicate that it is advisable to use a higher grade material.

EFFECT OF SOIL AND FERTILIZERS ON THE CHEMICAL COMPOSITION OF THE CANE.

The principal effect which the soil has upon the crop is shown in the quantity which is produced. It is well known, however, that the character of the soil also influences to a greater or less extent the chemical composition of the crop. Crops which are grown in a very fertile soil are usually coarser in texture than those grown in one less fertile. If, therefore, we are seeking for a particular flavor, taste, or character of product it is to be remembered that the fertility of the soil has an influence of quite a marked character in many of these particulars. If we examine the quantity of sugar contained in the cane grown on plat No. 24 of Field A, we find that the juice of the cane contained 15.72 per cent of sucrose and 0.44 per cent of reducing sugar with a purity of 91.4 per cent. This typical composition of the juice is shown by all the crops on Field A. It is a remarkable showing of purity and high sucrose content with a low reducing sugar content. The quantity of sugar in these canes and the purity of their juices compare favorably with the data obtained from sugar canes grown under favorable conditions in the Tropics. In fact, it appears that the purity is, as a rule, higher than that of the juices of tropical sugar canes. The effect of the fertilizer on Field A is not very marked in so far as variations in chemical composition are concerned.

Twelve of the fertilized plats, viz, Nos. 1, 4, 5, 13, 14, 15, 17, 18, 20, 21, 22, and 23 have a higher content of sugar than plat No. 24, and the other eleven have a lower sugar content.

In regard to the purity of the juices, seven of the fertilized plats, viz, Nos. 1, 5, 14, 15, 18, 21, and 23, have a higher purity than plat No. 24, and the others a lower purity. In this enumeration plat No. 13 is excluded, since the number expressing the purity as recorded is evidently an error and the data are not at hand to ascertain the magnitude of the mistake.

In regard to reducing sugar, ten plats, viz, Nos. 2, 5, 6, 7, 8, 9, 12, 15, 16, and 19, have a larger amount than plat No. 24. Four have the same amount, viz, Nos. 3, 10, 22, and 23, and the others a smaller quantity.

It is seen that the general effect of the added fertilizers on the composition of the juices is unimportant. If we compare, on the other hand, the character of the canes in Field B with plat 24 of Field A, we see at once a marked inferiority in the juices. This is shown both in the lower content of sucrose, a higher content of reducing sugar, and a lower purity. Not one of the plats in Field B produced a juice having as high sugar content, as small a quantity of reducing sugar, or as high purity as the juice of the cane on plat 24 of Field A.

Inasmuch as the same quantities of fertilizers were added to Field A and Field B, the conclusion is evident that the inferior character of the cane grown on Field B was due to the comparative newness of the soil and to the influence of the heavy crop of velvet beans plowed under during the previous season. It is not to be inferred from the above that the chemical character of the canes grown on Field B was low; on the contrary, they are of quite an excellent composition, both in content of sugar and in purity of juice. The inferiority, therefore, is in their comparison with the canes grown on Field A. The logical conclusion to be drawn from this discussion is that the large excess of available nitrogen and organic nitrogenous matter in the soil, while tending to produce a large crop, also has a decided tendency to diminish the excellence and purity of the product. It would be quite logical to infer from the above data that the canes grown on Field A would make a better quality of sirup, having a brighter color, and bringing a larger price in the market than that yielded by the same weight of canes grown on Field B.

SUMMARY OF RESULTS.

(1) Velvet beans have a high value as a fertilizing agent, but this value reaches its maximum only when other essential elements of plant food other than nitrogen are applied in the fertilizer.

(2) In the application of fertilizing materials it is far better not to apply the whole amount at the time of or before planting, but to apply a part of the fertilizer at the time of planting and the rest at intervals during the growing season.

(3) In regard to the amount of the fertilizer to be applied to obtain the best financial results, it appears that 1,200 pounds of the normal fertilizing material is probably the best amount for Field A, while for Field B a larger amount of this fertilizer may be used. It is plain, however, in this case, that the increased amount of fertilizer could be more profitably secured by adding larger quantities of phosphoric acid and potash and a smaller quantity of the nitrogenous constituents. On Field A there was a profit in using 1,200 or even 2,000 pounds of the fertilizer per acre rather than 800 pounds, but on Field B there was no

profit in using 1,200 pounds rather than 800 pounds, and a slight profit in using 2,000 instead of 800. In general it may be said that on land which contains a high percentage of fertilizing materials, as found in Field B, it is not advisable to use over 800 or 1,000 pounds of normal fertilizer per acre, while on land possessing only a normal natural fertility, as in Field A, 1,200 to 1,500 pounds per acre of normal fertilizer may be used to advantage.

(4) It is evident that fertilizers such as phosphoric acid and potash which contain large quantities of fertilizing ingredients give better results than those which contain small quantities.

(5) Muriate of potash apparently gives somewhat better results than kainit, especially on Field A.

(6) Nitrate of soda, upon the whole, seems to be a more satisfactory source of nitrogen than cotton seed and cotton-seed meal at practically the same cost.

(7) Sodium nitrate, however, should be added in small quantities at a time and at rather frequent intervals to avoid loss by leaching during the heavy rains. Cotton seed is a more expensive fertilizer than sodium nitrate or cotton-seed meal.

(8) On soils represented by Field A more nitrogen is needed in the fertilizer in proportion to the potash and phosphoric acid than on Field B, and vice versa.

(9) It is evident that excessive amounts of any single fertilizing ingredient are inadvisable, because the plants can not assimilate the excess. The ration for a plant should be carefully balanced to meet the requirements, basing the character of the fertilizer on the amount of available plant food already in the soil, and adding to the fertilizer larger quantities of the elements missing in the soil and diminishing in the fertilizer those elements which are abundant therein.

It will be interesting also in this connection to note the conclusions reached by Mr. Roddenbery from a study of the data given in Table XII. In this case we have the conclusions deduced by a practical man of affairs illustrating the fact that such scientific data have an immediate and extremely practical value when placed in the hands of a thinking business man, though he may not be a scientist. Mr. Roddenbery's deductions are as follows:

CONCLUSIONS ON THE FERTILIZER EXPERIMENTS.

By W. B. RODDENBERY.

FIELD A.

(1) A high percentage of nitrogen assists germination and favors suckering. Compare plats Nos. 5, 7, 8, and 16 with 9, 10, 11, 13, and 24.

(2) When applied, however, in the furrow with seed cane, heavy fertilization with complete fertilizers prevents germination. See plat No. 6 as to stand.

(3) Nitrate of soda is a better source of nitrogen than cotton-seed meal or cotton seed. Compare plat No. 19 with Nos. 11, 18, 20, and 21.

(4) Cotton seed is not a profitable fertilizer for sugar cane. Compare plats Nos. 20 and 21 with Nos. 3, 7, 10, and 23. Cotton-seed meal gives $14 per acre more profit than the same value in cotton seed. Compare plat No. 18 with plat No. 20. (In all estimates of profits cane is valued at $4 per ton.) Nitrate of soda shows $12 per ton more profit than the same value in cotton-seed meal. Compare plat No. 19 with No. 18.

(5) Two applications of the fertilizer give the best results.

(6) Doubling potash shows no gain in tonnage over that given by the normal fertilizer, and the general trend of the experiments indicates about 50 pounds of actual potash per acre as being the most profitable amount for this soil.

(7) Doubling nitrogen shows a gain of 3 to 3½ tons per acre over the tonnage given by the normal fertilizer and about $9 per acre more profit. In general, the experiment indicates 65 pounds of nitrogen per acre as the most profitable proportion for this soil, the source of the nitrogen to be mainly nitrate of soda.

(8) Doubling phosphoric acid gives no gain over the tonnage given by the normal fertilizer, but for some unknown reason shows a decrease of 1½ tons per acre and a gain of only 3½ tons per acre over the "no phosphoric acid" plat, No. 14. In general the experiments point to 100 pounds per acre of phosphoric acid as a sufficient amount for this land.

(9) All the results indicate that the following formula will give the fertilizer most suitable for a land like Field A:

	Pounds.	
12.5 per cent acid phosphate	800	(100 pounds phosphoric acid = 7.7 per cent).
Cotton-seed meal	100	(55 pounds nitrogen = 4.2 per cent).
Nitrate of soda	300	
Muriate of potash	100	(50 pounds actual potash = 3.8 per cent).

FIELD B.

(1) No special effect on the stand was noted as a result of the different fertilizers, except that plat No. 6 had a very poor stand at first; but this was overcome later by heavy suckering, which was probably the result of a large application of nitrogen to this plat.

(2) No difference was noted due to the source of the nitrogen, nitrate of soda and cotton-seed meal showing the same tonnage.

(3) Cotton seed is not as good as the same value in cotton-seed meal. Compare plats Nos. 18 and 20.

(4) Two applications give the best results.

(5) Doubling the potash gave no gain over the normal potash ration, and the general trend of the experiments points to 50 pounds per acre of actual potash as being sufficient on a soil of this kind.

(6) Doubling the nitrogen gives the same tonnage as the "no nitrogen" plats. Compare plats Nos. 13 and 16, both of which give 4 tons less per acre than the normal ration. The general trend of the experiments points to the need of a very small application of nitrogen; possibly none would have been more profitable, but 20 pounds per acre is certainly sufficient.

(7) Doubling the acid phosphate gives a gain of 13 tons per acre over the "no acid phosphate" plat and a gain of 3 tons over the normal ration. In general the need of a larger application of phosphoric acid is shown, and probably about 200 pounds of this fertilizer per acre would be the most profitable proportion.

(8) All results indicate that the following formula for a fertilizer would give the best results on such land as Field A, on which a heavy growth of velvet beans had been grown and plowed under the previous year:

	Pounds.	
12.5 per cent acid phosphate....	1,600	(200 pounds phosphoric acid=11.1 per cent).
Nitrate of soda..................	100	(nitrogen=1 per cent).
Muriate of potash...............	100	(50 pounds actual potash=2.72 per cent).

REPORTS OF SPECIAL AGENTS ON THE SUGAR INDUSTRY.

INTRODUCTION.

By G. L. Spencer, *Chief of Sugar Laboratory.*

There is probably not a State, Territory, nor possession of the United States, with the exception of Alaska, in which sirup, or a semisolid product, formed of a mixture of sugar and sirup, is not manufactured. In the Northern sirup industry this product is obtained from sorghum cane and the sap of the maple tree. In the South both sorghum and sugar cane are used in sirup manufacture. An additional source of sirup is maize or indian corn, from which large quantities of glucose are made. This branch of the sirup industry and maple sirup will not be considered in this bulletin.

The extent of the sirup industry is indicated by the following data from Bulletin No. 237, Agriculture, Twelfth Census:

> Of the total area devoted to sugar cane, exclusive of Hawaii, the South Atlantic division contained 47,223 acres, or 12.2 per cent, of which 55.2 per cent was in Georgia, and the South Central division 339,713 acres, or 87.8 per cent of the total, of which Louisiana contributed 81.5 per cent. There were 50 acres reported from Arizona in the Western division. The increase in area since 1889 in the South Atlantic division was 43.6 per cent, and in the South Central division 40.3 per cent.

The above figures indicate that comparatively little cane is grown for sirup making except in Georgia, Alabama, Florida, and Mississippi, since the cane in Louisiana and Texas is almost exclusively employed in sugar manufacture.

The increase in the South Atlantic division of 43.6 per cent is largely due to the expansion of the industry in Georgia. The Census report cited gives the total number of gallons of sirup produced as 12,293,032, the greater part of which was consumed as table sirup. A considerable quantity of sirup is produced in Louisiana and sold to the sugar factories. There are no data in the Census report to show whether this sirup is excluded from the returns for table sirup, but in any event these figures indicate the present magnitude of the cane-sirup industry. The Census report gives the following additional data in regard to sorghum sirup:

> In 1899, 446,621 farmers produced from 293,152 acres 1,911,046 tons of sorghum cane. Of this they sold 291,703 tons and from the remaining product manufactured 16,972,783 gallons of sirup.
>
> Of the total area devoted to sorghum, the North Atlantic division reported 126 acres and the South Atlantic 54,152 acres, or 18.5 per cent of the whole, of which Georgia and North Carolina together contributed 69.4 per cent.

The North Central division contained 92,166 acres, or 31.4 per cent of the whole, of which Kansas and Missouri together contributed 56.1 per cent. In the South Central division there were 145,812 acres, or 49.7 per cent of the whole, of which Alabama, Arkansas, Kentucky, Mississippi, Tennessee, and Texas together furnished 88.1 per cent. The Western division contained 896 acres, or 0.3 per cent ol the whole, of which 371 acres were in Utah. The North Atlantic division contained 0.1 per cent of the total acreage.

There was a decrease in area since 1889 of 29.5 per cent.

From these figures, showing a total production of table sirup amounting to 29,265,815 gallons from cane grown by more than 500,000 farmers, the importance of this industry and its extent are apparent. Louisiana has long been a producer of a favorably-known sirup termed "open-kettle molasses," while Georgia, Florida, and other Gulf States have lands upon which cane can be grown that produces sirup of exceptionally good flavor. The agricultural phase of the industry as it is now being developed in this section of the country is illustrated by the following reports of the special agents who have during the past year investigated the agricultural conditions and methods now existing.

SUGAR-CANE CULTURE AT CAIRO, GA.

By Ralph Hoagland and R. D. Stubbs, *Special Agents*.

Sirup making from sugar cane is the important industry in this section, there being numerous steam factories and a great many horsepower mills using the open-kettle process. During last season, October 1, 1901, to March 1, 1902, 10,777 barrels of sirup were shipped from Cairo (Plant System), while over the whole of that railway system in Georgia only 16,618 barrels were shipped, thus showing Cairo to be the center of the greatest sirup-producing section in the State.

In consultation with Mr. J. B. Wight, president of the Cane Growers' Association of Cairo, the following points were decided upon for study and investigation:

(1) Best fertilizer for tonnage of cane.
(2) Best fertilizer for sucrose content.
(3) Effect of potash in effecting early development of sucrose.
(4) Best method of application of fertilizers.
(5) Amount of fertilizer that can be profitably used per acre.
(6) Difference in sucrose content at time of full development, of cane fertilized with acid phosphate and potash and ammonia in excess, and of cane fertilized with normal fertilizer.
(7) Careful noting of any differences that may appear in any respect in cane raised on velvet-bean land as compared with cane on potato land.
(8) Differences in flavor of sirup made from cane receiving different fertilizers.
(9) Differences in purity coefficients of cane receiving different fertilizers.
(10) Determination, by measurement and weighing, of tonnage per acre in various fields to obtain the average yield per acre. (2) To determine maximum yield.
(11) Average juice extraction at various mills of different types.
(12) Fall planting of cane.
(13) Planting of tops.
(14) Best method of saving stubble.
(15) Variety tests, and breeding of cane to increase sucrose content.
(16) Best and simplest method of clarification for small factories; for steam factories.
(17) Effect of sulphur and lime upon quality of sirup.

The comments on the fertilizer experiments are found in Part I of this bulletin, even one year's work being very instructive as to some points, while in regard to others, such as methods of planting, cultivation, saving seed, etc., several years' work would be necessary to obtain conclusive results. The following general observations, however, on these points may be of interest:

Nature of the land.—The land is cut-over pine land, of which the surface soil is a sandy loam and the subsoil a red clay. The water supply is quite near the surface and the land rather level, but rolling enough to give good drainage. The natural fertility of the land is low and fertilizers are needed for all crops, even on new land. Fields which have been long in cultivation and have received little or no organic manures do not respond readily to heavy applications of commercial fertilizers, thus showing the need of humus in the soil.

Varieties of cane planted.—The common purple cane is the only variety planted extensively here for commercial purposes. Some green and ribbon cane is grown, but it is not as well liked as the purple. Mr. Wight, of Cairo, in 1902, planted two-thirds of an acre of cane known as No. 74, sent to him by Dr. W. C. Stubbs, of the Louisiana Sugar Experiment Station. This variety has a dark green color, growing very erect, but showing in this experiment no increase in tonnage over purple cane and a decrease in sucrose. The sirup produced from this cane, while clear and light in color, had such a salty flavor that it was not deemed advisable to can it. The cane was planted on high, dry land, but has a taste as though grown around horse lots, which was not the case.

Dr. Stubbs in writing in regard to the experiment with this cane said:

> No. 74 is one of our seedling canes, grown very successfully in the State. It is of early maturity, fine tonnage, rich in sucrose, a good soft cane, giving an extraction in our 9-roller mills of 80 per cent. I would not, by any means, take the first determinations you have made to be conclusive in regard to the value of this cane in your section. I believe it will have by the end of the season a much better showing, and on account of its excellent properties, besides its larger sucrose content, it is a cane greatly to be desired.

Experiments with this variety of cane will be continued another year. In comparing the Georgia and Louisiana cane, Dr. Stubbs said further:

> Your canes in Georgia are richer than ours here, and while the tonnage is much less, the manufacturer has less difficulty in sugarhouses in evaporating the juice. When sugar returns to its former value of 4 or 5 cents a pound, Georgia will unquestionably figure in manufacturing sugar.

A stalk of ribbon cane 11 feet 10 inches long was brought into Cairo on December 9, 1902, by M. J. Harrison.

Preparation of land, planting, suckering, etc.—The preparation of the land for planting is begun in December, immediately after the harvesting season. Two and three horse plows are generally used for loosening the soil, though sometimes harrows or disk plows are preferred. After the land is thoroughly broken to a depth of about 6 inches it is bedded into rows of good height, 4 or 5 feet apart, thereby securing satisfactory drainage and easy cultivation. The middles are then broken and beds opened deep with a long scooter, when the ground is ready to receive the cane, which is planted about April 1, one running stalk to the row. The cane is not covered deep, only 2 or 3 inches of soil being thrown over it. The row is raked to break the crust, if any be formed, which offers resistance to the young plants. When the cane sprouts a scooter furrow is run over each side, to aid in the development of suckers. No soil is thrown back until there is a sufficient number of suckers to insure a full stand, when it is sided with sweeps. The question of suckering is very important, and presents opportunity for further investigation as to the best time to plant cane in order to get the largest number of suckers, and how to cultivate them that they may be vigorous and develop quickly. The remaining cultivation is effected with a weeding hoe, and about July 1 the cane is laid by with five furrows to the row with a broad sweep.

Fertilization.—Fertilization is necessary to the growth of sugar cane in this section, and it is the most complex and perplexing problem which confronts the planter of southern Georgia. Various points must be considered in selecting a fertilizer, among them the resulting tonnage, sucrose content, and quality of product, in connection with the cost of the fertilizer. For instance, a fertilizer giving a very large yield might give a small sucrose content, and vice versa. It must also be determined whether the soil most needs phosphoric acid, nitrogen, or potash, what amounts of each will be most profitable, and what is the best time and method for applying the fertilizer. It is well known among fruit growers that potash promotes the early ripening of fruits, and the question arises as to whether the analogy would hold good for sugar cane, causing an early development of sucrose. A number of experiments along these lines, made at Mr. Roddenbery's plantation, are given in detail in Part I of this bulletin. In general, however, far too little attention is paid to the question. The fertilizer commonly used per acre is from 15 to 25 bushels of cotton seed and 800 pounds of guano, having about the following composition: 9 per cent of phosphoric acid, 2 per cent of nitrogen, 3 per cent of potash. Large amounts of cotton seed are used by nearly all the planters, some using as much as 90 bushels to the acre, although 25 bushels is the usual amount. The fertilizer is put on in two applications—one in the furrow with the cane and the other in drills at each side of the row later on in the summer. In using cottonseed and guano the cottonseed and half the guano are applied in the drill with the seed cane, while the remainder of the guano is applied in drills later in the season. Barnyard manure or compost is highly prized as a fertilizer for cane, and gives much larger returns than its mere fertilizing constituents could produce.

It is not only necessary that fertilizers be used during the cultivation of cane, but some form of humus must be added to the soil continually in order to prevent a decrease in the fertility. Two legumes are used for increasing fertility, viz, velvet beans and cow peas. Velvet bean land showed a distinct superiority over rye land with the same amount of fertilizer on both, and when plowed under as a green manure or pastured off by stock the beans undoubtedly have a very beneficial effect upon the land. Besides adding nitrogen to the soil, the beans probably help to make inert plant food available through the addition of humus to the soil, as does barnyard manure. The question arises, however, as to whether cow peas can not be more profitably grown for this purpose, although the velvet beans may add somewhat more fertility to the soil. The beans have a very rank growth, and can be used for feeding purposes only by grazing, causing of course a large waste, whereas if peas are planted together with corn, after they have performed their function of supplying nitrogen to the soil, the farmer gathers enough to plant next year's crop, and probably 15 bushels of corn per acre. Or the peas may be sown broadcast and mowed, making the finest hay obtainable. The question of obtaining food for stock confronts the farmers of this section, as the land naturally furnishes very poor pastures. For this reason velvet beans are used on only a few of the extensive plantations, and corn and peas are generally planted for an "upbuilder."

In regard to the question of cottonseed versus cottonseed meal, which is touched upon under fertilizer experiments, Mr. Wight, of Cairo, thinks that if cottonseed meal is used correctly, in proper proportion and properly applied, better results can be obtained than by using cottonseed. Good results have been obtained, however, by using the seed as a top dressing in middles when cane is laid by, the general opinion among the farmers being that cottonseed makes just as good a manure after sprouting as before. A disadvantage in using a large amount of seed is that the cane is frequently attacked by wood lice. This can be prevented, however, by mixing the seed with guano containing phosphoric acid before applying. The usual amount of cottonseed applied is 15 bushels per acre, while of cottonseed meal 400 bushels per acre is used.

While drainage is not a question with the south Georgia planter, the best cultivation during a drought is a practical issue, and here again fertilization must be considered, for oftentimes a fertilizer that would make the best cane if there were plenty of rain would ruin the crop if dry weather prevailed.

Harvesting of cane.—Sirup making usually begins during the latter part of October. This year (1902) grinding generally began on October 27, although some small mills started a week or ten days earlier. Cutting begins when the juice tests about 8° Baumé or when most of the leaves of the cane, except the tuft at the top, are dead. The cane is stripped a short time before cutting, topped and cut close to the ground with a broad, heavy hoe. The patent stripper is very satisfactory when the cane is fairly straight. The implement generally used in this section is a cane stripper which has a flattened handle about 3½ feet long, to one end of which a steel spring is attached by means of a slide bolt. The cane is pushed through the opening into the oval portion of the spring and the implement pushed down the stalk, thus removing the leaves.

The top, above the first red joint in the case of the purple cane, is taken off with a corn knife just before the cane is cut. The farmers in this vicinity make no use of the tops, though they have been tried for seed and given good results, the cane from them giving just as large a tonnage and as much sirup as cane from stubble.

The cane is cut only a short time before hauling to the mill, very little being cut ahead. Negroes strip, top, cut, and pile up the cane at convenient distances apart on every third row for from $3.75 to $4.50 per acre. An ordinary wagon is used for hauling, and two large hemp ropes are laid in the wagon and the cane placed straight upon them. The load being procured, the ropes are tied, and thus you have two large bundles of cane on the wagon, which are lifted off by machinery at the mill and placed in position. Mr. T. Wight's two-horse wagons generally haul from 1,200 to 2,000 pounds of cane per load.

Cane that is stripped but not topped and left standing in the field does not increase in sucrose any more rapidly than cane that is not stripped. To try the effects of stripping and topping the cane and of leaving it unstripped and untopped before cutting, samples for analysis were taken under the following conditions:

A. Samples of cane stripped ten days before cutting, and topped when cut.

B. Samples of cane topped and stripped ten days before cutting, from the same plat as sample A.

C. Sample of cane stripped and topped when cut from the same plat as sample A.

B 3. Samples of cane stripped and topped when cut, to replace sample C, which was not a good average sample.

The canes from which the different experiments were made were harvested at Cairo, Ga., on November 11, 1902, and analyzed as soon as they reached Washington, D. C. The analytical data obtained on these samples are as follows:

TABLE XIII.—*Analyses of canes harvested under varying conditions.*

Sample.	Sucrose.	Purity.	Reducing sugar.
	Per cent.		*Per cent.*
A.	11.85	80.6	1.56
B.	11.85	81.7	1.21
C.	13.63	83.1	1.38
B 3.	12.56	79	1.94

From these data it is seen that it is not a good policy to strip or top the cane before cutting. Both samples A and B were inferior to samples C and B 3, and markedly inferior to C. Sample C, however, is not regarded as a typical sample. Samples A and B were superior to B 3 in having a smaller content of reducing sugar and a higher purity. This superiority, however, is only from a chemical point of view and not from a sirup-making standpoint. In other words, a sirup made from sample B 3 would be less likely to granulate than a sirup from samples A and B. The total quality of sirup-making materials is obtained by adding together the sucrose and the reducing sugar. Judging from these tests, samples C and B 3 are positively superior to A and B

Saving stubble for seed cane.—No cane is planted in the fall in this vicinity, as first-year stubble usually supplies sufficient seed cane. Many planters do nothing to save stubble and hence first-year stubble cane is very uncertain, and at best but a poor crop. Some planters, however, save the seed cane in the following way: The cane is dug up by the roots during the latter part of October, just before the grinding season begins, placed in large windrows 4 to 5 feet wide and 2 to 3 feet deep, the tops of the cane lapping the butts and covered with a few inches of dirt from two or three furrows thrown up on each side of the windrow. Cane so stored in well-drained land keeps very well through the winter. But very little first year planted cane is used for seed. In saving seed cane it is calculated that 1 acre of good cane will plant 6 acres, or if the cane is short, 5 acres.

Data on Mr. Polk's farm.—Mr. Polk has 10 acres of excellent purple cane, about as good a stand as was seen. The rows were 5 feet apart and the cane tall and heavy, but rather green. The land has been in cultivation twenty years, but has been well manured. The fertilizer is put on in two applications, one in the drill at planting and one in June, and has the following composition: Thirty bushels of cotton seed per acre; 400 pounds of guano (1.88 per cent nitrogen, 11 per cent soluble phosphoric acid, 2 per cent insoluble; 1 per cent of potash). This fertilizer costs about $22 per ton retail, or $19 wholesale. In addition 4 loads of stable manure per acre were used. Mr. Polk values manure very highly and probably the fine condition of the cane was largely due to its use. In saving seed cane furrows are plowed from each side over the stubble after a part of the waste has been burnt off, and very good results have been obtained.

Data on the farm of Mr. Vanlandingham.—Three and three-quarters acres of purple cane were planted on March 1, 1902, from which a yield of 69.2 tons was obtained, an average of 18.5 tons per acre.

The fertilizer used per acre was 600 pounds of commercial guano (8 per cent phosphoric acid, 4 per cent potash) and 15 bushels of cotton seed. The land was broken broadcast and bedded into rows 4½ feet wide, in which deep furrows were opened and the cane put down with 400 pounds of the guano mentioned above and 15 bushels of cotton seed. The seed were sprinkled down "green" in the drill when the cane was planted. It is then lightly covered, unless the season is very dry, when it is covered deep. The row is run over with a board, or better with a rake, thus leveling off the uneven spots. The cane is plowed only twice, barred off with a sweep, and no soil is thrown back until a full stand of suckers is obtained. The cane is thinned and laid by when shoulder high to a man of medium height, by plowing five furrows to the row with a large sweep. This is generally done about July 1, and Mr. Vanlandingham thinks that cultivating after this time does more harm than good, as it injures the feed roots.

The crop was sold to Mr. Wight, of Cairo, for $3.75 per ton. Immediately before harvesting cane for planting is dug up with tops and roots and placed in windrows 4 to 5 feet wide with butts 8 inches apart and tops overlapping to keep the dirt from them. The soil is thrown on to a depth not exceeding 2½ inches, as Mr. Vanlandingham thinks more cane is lost in this section from heat than from cold, the cane being covered too deep in the windrows. When the cane is dug up for planting it is stripped, topped, and cut in two pieces, so that if a piece is pulled up in cultivating less is displaced.

The first killing frost occurred on November 29, and as there had not been enough frost previous to that date to stop the growing of the cane, the stubble had sprouted and was killed, leaving only the eyes underground to provide the stubble cane of the next season.

Data from the farm of Mr. J. B. Wight.—Mr. Wight planted about 33 acres with purple cane between March 15 and April 1. The cultivation was much the same as that already described, the fertilizer used being as follows:

> Eight hundred pounds of guano (9 per cent phosphoric acid, 2 per cent of ammonia, and 3 per cent of potash), at $19 per ton; 300 pounds of cotton-seed meal, at $23.40 per ton; 15 bushels of cotton seed, at 25 cents per bushel. Total cost per acre, $14.86.

Four hundred pounds of guano and 15 bushels of cotton seed were put in the drill when the cane was planted; 400 pounds were put in siding furrows, and 300 pounds of meal in the middle when the cane was laid by.

The following estimates as to the profits on raising sugar cane for sirup making in southern Georgia, involving the consideration of the advisability of having one central mill for each community fitted out with the most modern machinery procurable, are taken from an address made by Mr. Wight before the fourth annual convention of the Association of Commissioners of Agriculture of the Cotton Growing States, held at Nashville, Tenn., in August, 1902:

Approximate cost of bringing 1 acre of sugar cane to maturity and manufacturing it into sirup.

Seed cane to plant 1 acre	$10.00
Fertilizer	10.00
Cultivation and rent of land	10.00
Stripping and hauling	10.00
Manufacturing into sirup	20.00
Thirteen empty barrels, at $1.10 each	14.30
Total cost	74.30
Thirteen barrels of sirup (32 gallons per barrel), at 25 cents per gallon	104.00
Profit per acre	$29.70

In case the farmer has no mill and gets a neighbor to make his sirup, the cane grower delivers the cane at the mill, the mill owner bears all the expense of manufacturing, each pays for the barrel to hold his share of the sirup, and the owner receives three-fourths of the product and the manufacturer the remainder. In such a case the account of the grower stands thus:

Cost of growing and delivering cane at mill	40.00
9¾ empty barrels	10.73
Total cost	50.73
9¾ barrels (312 gallons) of sirup, at 25 cents per gallon	78.00
Net profit to grower per acre	27.27

A comparison of these figures with those of other field crops of this section shows sugar cane to have the advantage. It is also to be noted that the figures given represent only an average crop, sometimes as much as 800 gallons of sirup per acre being obtained.

In the States east of Louisiana 99 per cent of cane is manufactured into sirup, which brings up the question of overproduction. But when 90 per cent of the sirup sold in grocery stores is an inferior and adulterated product and the demand for a good quality of sirup is increasing, no fear from this source need be entertained. In regard to the increased demand for good sirup which has been developed at Cairo, Mr. Wight says:

> Sixteen years ago it was hard to find a market for the 1,500 barrels of sirup annually shipped from the town, and one crop was frequently not sold before another was produced. Now that a better grade of goods is being made, 12,000 barrels of sirup are easily sold before another crop is produced. This sirup is marketed from Massachusetts to Texas, 3,644 barrels of last year's crop going from Cairo to the latter State alone.

As would be expected from the known effect of the quality and grade of land on the sucrose content of cane, the sirup produced from cane grown on the higher lighter lands of South Carolina, Georgia, Alabama, Mississippi, and Florida is of a better quality than that produced on the low alluvial lands of Louisiana.

In regard to sugar cane versus sugar beets the following data are given:

The cost of growing an acre of sugar beets, exclusive of the rent of the land, averages $30. The average yield is 10 tons per acre, which sell at from $4 to $4.50 per ton. This gives gross returns varying between $40 and $54 and a net profit of from $10 to $24 per acre. These figures give sugar cane an advantage of from $8 to $9 per acre over beets.

The cane in 1902, until about November 20, was not as sweet as usual, because the autumn was late and exceedingly warm and the cane continued to grow and therefore matured very slowly. This was a disadvantage to farmers having a large acreage, as they could not wait until the most suitable time for making sirup because the limited capacity of the mills would in such a case prolong the time of manufacturing too far.

The following table shows the composition of the cane juices as obtained at the factories of J. B. and T. Wight:

TABLE XIV.—*Composition of cane juices sampled at the factories of J. B. and T. Wight, Cairo, Ga.*

Proprietor.	Date of observation.	Composition of juice.		
		Sucrose.	Reducing sugars.	Purity.
	1902.	*Per ct.*	*Per ct.*	
T. Wight	Oct. 28	10.06	2.23	76.82
J. B. Wight	Oct. 29	8.95	1.93	69.92
T. Wight	...do...	10.27	1.55	75.44
Do	Oct. 30	10.22	2.11	74.14
Do	Oct. 31	10.36	1.99	72.96
Do	Nov. 1	10.43	1.90	77.26
Do	Nov. 3	10.28	2.25	71.40
Do	Nov. 5	11.30	1.82	78.47
Do	Nov. 6	Rejected.		
J. B. Wight	Nov. 7	9.22	1.58	70.38
Do	...do...	11.55	1.51	77.51
Do	Nov. 10	11.12	1.81	76.68
T. Wight	Nov. 11	11.88	1.78	79.20
Do	Nov. 12	10.82	1.82	73.10
Do	Nov. 13	11.06	1.52	76.28
T. Wight	Nov. 14	12.15	2.02	82.64
Do	Nov. 17	11.83	1.92	75.84
Do	Nov. 18	11.28	1.81	75.69
J. B. Wight	Nov. 19	11.77	1.15	79.52
Do	...do...	12.61	1.59	81.88
T. Wight	...do...	11.75	1.63	77.31
Do	Nov. 20	12.19	2.19	75.25
Do	Nov. 21	12.21	1.69	79.80
Do	Nov. 24	13.18	1.15	82.38
Do	Nov. 25	13.31	.72	83.71
Do	Nov. 26	12.83	1.27	82.77
Do	Nov. 27	13.15	.92	83.22
Do	Nov. 28	12.64	1.44	82.62
Average		11.42	1.68	77.49

CANE GROWING IN THE VICINITY OF GUYTON, GA.

By ARTHUR GIVEN, *Special Agent.*

The country about Guyton is very well adapted to cane growing. It is flat, with a slight slope toward the Ogeechee River. The soil is a rich black loam with clay subsoil at a depth of from 8 inches to 2 feet. Some of the land is rather high, with light soil, and does not produce large cane crops. The lower land requires to be drained as in Louisiana. South and east of Guyton is a great extent of swamplands which, if cleared and drained, would make valuable cane land. Every farmer in this part of the State grows from one-quarter to 2 or 3 acres of cane, and some of them as much as from 5 to 20 acres. The largest of the mills in this vicinity is that of Mr. J. T. Wells, where cane juices were sampled for the analyses given in the following table. The cane was from farms near Guyton and from two plantations at Halcyon Dale and Pooler, 10 and 14 miles distant, respectively, from Guyton by rail.

Table XV.—*Composition of sugar-cane juices sampled at the factory of J. T. Wells, Guyton, Ga.*

Date of observation.	Composition of juice.			Date of observation.	Composition of juice.		
	Sucrose.	Reducing sugars.	Purity.		Sucrose.	Reducing sugars.	Purity.
1902.	*Per cent.*	*Per cent.*		1902.	*Per cent.*	*Per cent.*	
Nov. 20	11.47	1.97	76.97	Dec. 13	9.43	2.86	64.14
24	9.68	3.12	66.75	15	10.39	2.14	72.16
28	9.99	2.01	68.89	16	9.95	2.37	72.63
29	9.18	2.40	66.52	17	8.99	2,86	62.42
Dec. 1	10.44	1.74	74.05	18	9.50	2.83	62.08
2	11.23	1.40	77.44	19	11.08	2.02	72.42
2	9.68	2.42	71.18	22	10.26	2.56	62.94
2	10.04	2.32	70.70	23	9.82	2.76	62,54
2	9.68	2.42	69.64	27	10.50	3.18	64.08
2	10.39	2.42	69.27	29	9.69	3.23	60.18
3	10.96	1.52	76.64	30	12.00	1.64	74.58
4	11.26	1.32	77.65	31	10.16	2.30	68.18
5	13.07	1.48	81.69	1903.			
8	13.37	1.29	81.52	Jan. 2	9.01	2.88	67.24
9	12.08	1.34	82.58	5–6	9.42	2.78	65.86
10	11.51	1.39	78.29	Average	10.50	2.24	70.69
11	11.52	2.28	71.56				
12	10.18	2.56	69.25				

CANE GROWING FOR SIRUP MAKING AT WAYCROSS, GA.

By Harold A. Ives, *Special Agent.*

Sugar cane is the most important crop grown in this vicinity and will be planted next year to a greater extent than ever before. The soil here seems to be perfectly adapted to the successful raising of the several varieties of sugar cane—purple, green, and ribbon—the purple cane, however, being more extensively used than the other two varieties. Cotton seed and cotton-seed meal are the fertilizers most used, although when available cow-pen fertilizer has the preference. The plan followed by most of the farmers is to fertilize before planting and again when the plants stand about 3 feet above the ground.

The sugar cane when planted for seed is frequently injured by a small worm which eats all the way through the stalk, and if the next stalk lies against the first the worm goes on through both. In order to prevent this the cane is cut into short pieces of about six joints and planted so that the end of one piece is not on the same line with that of another.

The cane is planted in rows about 4 feet apart, and it requires about 2,000 average-sized stalks to plant an acre. Before and shortly after the cane makes its appearance the ground is kept plowed in order that the earth may be loose about the young plants. After this all that is necessary is to keep the weeds from crowding out the young cane. The plant will soon protect itself from the weeds and grass, and then no more cultivation is necessary.

Within a radius of 5 miles of Waycross there are about 450 acres of sugar cane available for manufacture. As a rule the farmers use about one-sixth of an acre to plant an acre—that is, 16⅔ per cent of their cane is used for seed. In 1902 the farmers used about 30 per cent of the crop for seed, as the cane was damaged by frost and was therefore small.

The following analyses show the composition of the cane juices from the various plantations about Waycross:

TABLE XVI.—*Composition of cane juices sampled at factories near Waycross, Ga.*

Proprietor.	Date of observation.	Composition of juice.		
		Sucrose.	Reducing sugars.	Purity.
	1902.	*Per ct.*	*Per ct.*	
E. M. Eunice	Nov. 17	16.79	0.30	86.10
W. J. Booth	....do...	13.66	1.29	80.83
L. Anthony	Nov. 21	10.68	2.09	64.33
T. J. Colson	Nov. 25	12.01	1.48	....
Do	....do...	8.96	3.81	63.10
J. L. Stephens	Nov. 26	15.29	1.06	89.95
S. F. Miller	....do...	10.49	3.07	73.36
J. J. Wilkinson	Nov. 28	10.79	2.11	67.02
N. G. Lang	....do...	9.60	2.37	67.13
W. S. Chancey	Dec. 4	15.08	.66	88.18
L. E. Miller	Dec. 8	16.55	0.43	....
Do	....do...	13.64	1.85	81.67
W. N. Keton	Dec. 10	14.57	.99	83.25
P. L. Hatcher	Dec. 11	10.20	2.55	70.34
Do	Dec. 12	11.32	2.25	70.34
W. H. Boatwight	Dec. 13	15.57	.50	....
Average	....	12.83	1.64	75.82

CANE GROWING IN THE VICINITY OF QUITMAN, GA.

By J. M. STARR, *Special Agent.*

Around Quitman cane seems to be grown only as an incidental crop. Most farmers who run from 3 to 10 plows plant from 4 to 8 acres of cane. The small farmer with one horse or mule plants from three-fourths acre to 1½ acres. There is a distinct tendency to increase the acreage planted around Quitman, and many farmers have expressed the intention of planting at least 40 per cent more in 1903 if the seed cane comes through the winter well. The farmer who grows cane on the largest scale is Mr. McRae, who lives about 12 miles from the town. It is estimated that the amount of sirup handled at Quitman for jobbing purposes will be approximately 4,500 barrels. The average price which the farmer receives is 25 cents per gallon.

Three kinds of cane are grown in this locality, viz, purple cane (two varieties, big short-joint cane, and small long-joint cane), ribbon cane (two varieties, red striped and green striped), and green cane. The purple cane is most extensively grown for sirup making, and the ribbon cane is the next in importance. The small red cane grows as tall as the ribbon variety, but does not stand so thick and is generally thought to be sweeter. The green cane is not much grown for sirup making. It is very soft and tender, and the seed cane does not keep so well through the winter as the other varieties.

Almost all farmers who plant several acres of cane use cotton seed and commercial fertilizers containing nitrogen. Many of the cane growers use fertilizers that contain too much ammonia, which results in red, strong sirup. In the freshly cleared land, where there is considerable decayed organic matter in the soil, acid phosphate is the principal fertilizer used. The small farmers who plant only about an acre or two of cane use cow-pen manure and cotton-seed meal. About 30 bushels of cotton seed are used per acre and from 400 to 800 pounds of commercial fertilizer. When cotton seed is applied it is used as bedding for cattle before planting time. A deep furrow is plowed, in which the fertilizer is placed. The seed cane is cut into pieces of three joints each and dropped into the row about 3 inches apart and covered with a lister. After the cane comes up some farmers put in an additional amount of fertilizer at the sides of the row, and when the cane is laid by another application is sometimes made in the middle of the row. When the fertilizer is put in the row before planting it is usually mixed with the soil by running a plow through it before the seed cane is planted. The cultivation is of a simple character, since as soon as the cane gets a start it shades the ground and prevents the growth of weeds and grasses.

The weight of the cane per acre on the farm of Mr. McIntosh, 9 miles north of Quitman, was determined on November 26. The cane from 1 acre weighed 61,215 pounds, equivalent to 30.6 short tons. The number of canes in 1 load were counted in order to determine the average weight of each cane. The load contained 960 canes and weighed 2,112 pounds, which will make the average cane weigh 2.2 pounds. In computing for 1 acre, this gives as the total number of canes 27,825 +. This weight of cane makes from 16 to 20 barrels of sirup, averaging 33 gallons each. It requires a mean of 7½ gallons of juice to make 1 gallon of sirup. The average product per acre in the vicinity of Quitman may be safely placed at 14 barrels of 33 gallons each per acre, which amounts to 462 gallons.

One of the principal items of expense in the sirup industry is the seed cane, since very little dependence can be placed in the stubble crop. One acre of cane will plant from 4 to 10 acres, according to the amount grown, but much depends on how the seed keeps through the winter. Freight rates from Quitman are very high also.

If the grower can find out how to make sirup that will not ferment he can hold his product until there is a good price for it. The dealers will then advertise and handle the goods twelve months in the year instead of three, and there will be a great increase in price and acreage. This would render the adulteration of sirup with glucose unprofitable.

The following analytical data were obtained on the samples collected at Quitman and in the vicinity:

TABLE XVII.—*Composition of cane juices sampled at Quitman, Ga.*

Proprietor.	Date of observation.	Composition of juice.			Proprietor.	Date of observation.	Composition of juice.		
		Sucrose.	Reducing sugars.	Purity.			Sucrose.	Reducing sugars.	Purity.
	1902.	*Per ct.*	*Per ct.*			1902.	*Per ct.*	*Per ct.*	
E. J. Young	Nov. 4	8.85	2.51	65.07	Mabbett Bros	Nov. 20	11.18	1.73	76.57
Avera Bros	...do	10.58	2.32	72.97	Avera Bros	Nov. 21	12.34	1.75	79.10
Do	Nov. 5	11.89	2.02	78.22	Do	Nov. 24	12.94	.80	73.94
Do	Nov. 6	10.24	1.75	76.98	Do	Nov. 25	13.39	1.04	84.74
Do	Nov. 7	11.42	1.89	76.63	Do	Nov. 28	14.33	.66	85.80
Do	Nov. 8	11.87	1.91	77.58	Do	Dec. 1	12.75	1.13	84.44
Do	Nov. 10	10.99	2.07	73.75	Do	Dec. 2	10	2.32	71.43
Do	Nov. 11	10.60	2.47	74.65	W. Renifroe	Dec. 11	12.41	1.35	80.59
Do	Nov. 12	11.33	1.94	77.07	Do	Dec. 12	12.48	.88	86.02
Do	Nov. 13	11.20	1.35	77.24	H. Turner	...do	13.04	1.19	82.01
Do	Nov. 14	11.20	2.05	76.19	Mr. Harrell	Dec. 15	16.71	.54	....
I. Mabbett	...do	11.41	1.94	77.62	W. Renifroe	Dec. 16	11.50	1.57	79.86
Avera Bros	Nov. 17	11.91	1.89	79.38	H. Turner	Dec. 18	11.83	1.62	76.33
Do	Nov. 18	12.37	1.89	77.80	Mr. Adams	Dec. 19	13.85	.77	81.87
Do	Nov. 19	12.18	1.35	78.58	Do	Dec. 20	12.15	1.24	77.38
E. J. Young	...do	11.74	1.55	79.86	Average	....	11.95	1.60	77.97
Avera Bros	Nov. 20	11.61	1.71	77.40					

CANE GROWING FOR SIRUP MAKING IN ALABAMA.

By THOMAS BRAGG, *Special Agent.*

The greater part of the observations in Alabama were made at the farm of Mr. George W. Black, near Geneva. The land on which the cane was grown is of the sandy type with a red clay subsoil, and has been in continual cultivation for about sixty years. The previous crops were principally cotton and corn. Thirty-five acres of the purple variety of cane were planted by Mr. Black, 10 acres being reserved and bedded for seed and no care whatever taken of the stubble.

The cane was planted March 1 and cultivated about the same as corn. It was fertilized with 50 bushels of cotton seed and 600 pounds of guano per acre. The cane was cut about December 1, piled in rows, and left in the field until grinding began, when it was stripped and hauled to the mill.

The yield of cane and sirup per acre could not be obtained, as there were no scales available at the factory. However, 6 acres of the cane land were measured off, and it was found they produced 1,980 gallons of sirup, or an average of 330 gallons per acre. The farmers in this section greatly prefer the sandy lands for cane, as they find that the sirup from the cane grown on sandy lands is lighter in color than that produced on the dark bottom lands, and also that the juice contains a larger per cent of sugar. It is also claimed that it is difficult to obtain a good stand of cane on the bottom lands owing to the cold, damp nature of the soil.

The purple variety of cane is better adapted to the conditions in this section than the other varieties. The farmers think that it is hardier and that while the ribbon and green varieties produce a lighter sirup than the purple cane, the yield of sirup is smaller.

The following table gives the analyses of juices from farms in the vicinity of Geneva:

TABLE XVIII.—*Composition of cane juices sampled in the vicinity of Geneva, Ala.*

Proprietor.	Post-office.	Date of observation.	Composition of juice.		
			Sucrose.	Reducing sugar.	Purity.
		1902.	*Per cent.*	*Per cent.*	
Mr. Tucker	Lafayette	Nov. 10	8.91	3.17	62.30
Do	do	Nov. 11	8.37	3.53	58.54
Selman factory	do	do	8.03	3.87	57.77
Do	do	Nov. 12	7.56	3.93	55.18
Mr. Cooper	Opelika	Nov. 14	11.03	2.88	72.10
Mr. Beasley	Auburn	Nov. 18	10.70	2.76	69.48
Mr. Culver	do	Dec. 1	14.39	1.09	87.20
Mr. Black	Geneva	Dec. 23	14.31	.89	87.25
Do	do	Dec. 30	13.34	.80	82.34
Do	do	Dec. 31	14.44	.87	88.04
		1903.			
Do	do	Jan. 1	13.50	1.10	83.01
Do	do	Jan. 3	13.82	.91	85.84
Do	do	Jan. 6	14.19	.99	88.69
Do	do	Jan. 7	13.58	.99	82.80
Do	do	Jan. 9	15.27	.68	89.30
Do	do	Jan. 10	14.77	.74	89.10
Average			12.26	1.73	77.43

These data show that the greater part of the cane juice sampled in Alabama, especially that from Geneva, is of a fine quality, having a comparatively low percentage of reducing sugar and a high purity. In this connection it must be remembered that the samples of juice were not representative of a single cane, but were taken from a large volume of expressed juice, which represented the regular mill juice. The smallest amount of juice from which a sample was taken was 600 gallons and the largest 1,600 gallons.

GROWING SUGAR CANE FOR SIRUP MAKING IN MISSISSIPPI.

By E. E. Hudson, *Special Agent.*

The making of cane sirup is not carried on extensively in this section of the country because there is no good market for sirup here. There are only two or three mills that make sirup to put on the market, and these are not very large ones, averaging about 800 gallons per day. The majority of the planters have their own mills and make sirup only for private use. The best quality of sirup made this season was from cane grown on sandy bottom land. This depends altogether, however, on the season. If it is a wet season the pine uplands produce the best quality of cane, but when the season is very dry the bottom lands give the best crop.

The varieties of cane planted here are the white and the red or Cuban cane. The white cane, however, is not very hardy and therefore is not planted very extensively. Some of the planters, if the winter is not very severe, allow their stubble to remain in the ground, covering it over with earth to preserve it, but if there is a hard freeze during the winter they plow the stubble up and plant seed cane.

Cotton seed and cotton-seed meal are used altogether for fertilizing the cane. Sometimes stable manure is used, but not often, as it is thought to detract greatly from the sweetness of the cane. The following table shows the quality of the juice sampled at Mississippi factories, together with the character of the land on which the cane was grown:

Table XIX.—*Composition of cane juices sampled in the vicinity of Magnolia, Miss., and character of soil.*

Proprietor.	Post-office.	Date of observation.	Composition of juice.			Character of soil.
			Sucrose.	Reducing sugar.	Purity.	
		1902.	*Per ct.*	*Per ct.*		
Coney Sirup Works	Magnolia	Oct. 29	12.94	1.44	85.14	Upland sandy loam (drought).
E. J. Hurst	do	Oct. 30	12.18	1.27	74.26	Upland.
D. G. Lambert	do	Oct. 31	10.86	1.94	67.04	Black sandy loam; made land.
W. J. Lenore	do	Nov. 4	9.62	3.66	61.27	Upland.
V. F. Coney	do	Nov. 5	9.44	2.28	75.52	Pine upland.
W. Brint	do	Nov. 7	12.77	1.72	80.32	Light sandy loam.
Fernwood Sirup Works	do	Nov. 8	9.87	2	82.25	Pine upland; highly fertilized.
Lampton Sirup Co.	do	Nov. 10	11.69	1.99	75.42	Black sandy soil; stiff red clay subsoil.
J. A. Johnston	do	Nov. 11	12.96	1.62	80	Sandy, bottom land.
N. McGee	do	Nov. 12	15.51	1.19	....	Sandy, bottom loam.
F. M. Lee	Walkers Bridge	Nov. 13	11.51	2.19	76.23	Do.
C. W. Lang	Magnolia	Nov. 14	11.51	1.60	72.40	Pine upland.
J. A. Cutraer	do	Nov. 15	13.75	1.81	89.29	Bottom river land.
Klondike Farm	do	Nov. 17	8.70	1.64	57.24	Upland, sandy loam.
H. Dunnerway	Fernwood	Nov. 18	11.83	2.25	75.85	Bottom land, sandy loam.
J. A. Cutraer	Magnolia	Nov. 19	11.84	2	72.65	Pine upland.
Bridges Sirup Works	Bridges	Nov. 21	13.59	1.48	81.86	Bottom land, sandy loam.
A. Fitzgerald	Fernwood	Nov. 24	12.10	1.97	77.56	Pine upland.
J. B. Slade	Magnolia	Nov. 25	15.21	.61	87.91	Very high, sandy, pine upland.

TABLE XIX.—*Composition of cane juices sampled in the vicinity of Magnolia, Miss., and character of soil*—Continued.

Proprietor.	Post-office.	Date of observation.	Composition of juice.			Character of soil
			Sucrose.	Reducing sugar.	Purity.	
		1902.	*Per ct.*	*Per ct.*		
H. Prescott	Magnolia	Nov. 26	11.28	1.29	64.46	Pine upland, clay loam.
R. M. Frith	do	Nov. 29	13.96	.63	85.64	Sandy hill land, clay subsoil.
D. F. Vaughn	do	Dec. 3	11.64	.86	75.82	Bottom land, sandy loam.
E. J. Hurst	do	Dec. 4	11.65	.89	73.74	Do.
Allen Bros	do	Dec. 5	15.39	.84	....	Do.
H. O. Allen	do	Dec. 8	15.49	.84	....	Do.
Allen Bros	do	Dec. 9	17.62	.31	....	Do.
Simmons Bros	do	Dec. 10	17.93	.30	....	Do.
D. G. Lambert	do	Dec. 11	14.07	.74	86.32	Pine upland.
Average			12.75	1.46	76.41	

GROWING SUGAR CANE FOR SIRUP MAKING IN FLORIDA.

By GEORGE E. WALKER, *Special Agent.*

Much Japanese cane is being planted in the vicinity of Huntington, Fla., and it is highly praised, but none of it has been ground as yet. The interest in cane growing in this locality is increasing, and a much larger acreage would be planted in cane were it not for the difficulty of fencing out the wild hogs.

In consequence of dry weather in the early spring a good stand of cane was not obtained on the high pine land, and this fact, combined with the depredations of hogs running at large, left only enough cane for the seed requirements on our own farm, and consequently we were dependent on the outside supply to meet the demands of the factory. The demand, however, for seed cane (for Georgia principally) was so great that it paid better to sell seed cane than to make it into sirup. For this reason many carloads of cane were shipped from Huntington which it had been expected would go to the local factory, and thus the successful operation of the mill was rendered impossible.

The following table shows the composition of the juice extracted from cane grown at Huntington and at Kissimmee:

TABLE XX.—*Composition of cane juices sampled in Florida.*

Proprietor.	Post-office.	Date of observation.	Composition of juice.		
			Sucrose.	Reducing sugar.	Purity.
		1902.	*Per cent.*	*Per cent.*	
I. Braddock	Huntington	Dec. 30	11.94	1.39	76.05
Do	do	Dec. 31	13.10	1.41	76.17
Average			12.52	1.40	76.11
		1903.			
S. L. Lupfer	Kissimmee	Apr. 4	17.30	.24	88.70
Do	do	do	16.60	.36	89.50
Average			17.00	.30	89.10

SUMMARY.

Collecting the averages of each set of analyses the comparison shown in Table XXI is obtained.

TABLE XXI.—*Summary of analytical data on cane juices.*

Town and State.	Composition of juice.			Number of samples.
	Sucrose.	Reducing sugar.	Purity.	
	Per cent.	*Per cent.*		
Cairo, Ga	11.42	1.68	77.49	27
Guyton, Ga	10.50	2.24	70.69	32
Waycross, Ga	12.83	1.64	75.82	16
Quitman, Ga	11.95	1.60	77.97	32
Geneva, Ala	12.26	1.73	77.43	16
Magnolia, Miss	12.75	1.46	76.41	28
Huntington, Fla	12.52	1.40	76.11	2
Kissimmee, Fla.[a]	17.00	.30	89.10	2
Average	12.03	1.82	75.99	....

[a] Excluded from average.

From the above averages it is seen that the canes have a fairly constant composition in all of the localities where investigations were made. The canes at Guyton had the lowest content of sucrose, the highest content of reducing sugar, and the lowest purity; all the others run very close together. A comparison of the other analyses with the samples from Kissimmee, Fla., is not permissible. Only two of the samples sent from Florida (Huntington) were mill juices. The average of these two, as will be seen by referring to the table, agrees very closely with that of the samples from the other localities. At the beginning of April, 1903, two samples of canes were sent from Kissimmee, which had been growing all winter untouched by frost. The sucrose in these samples had increased about 4 per cent and the reducing sugars were only about one-fifth as great as in the Huntington samples taken earlier in the season. The purities were 13 points higher than those obtained during the milling season.

The data in general are most instructive in showing the average composition of the canes of the localities inspected. When special canes are selected, as has been the case in previous years, the analyses naturally show very much better results. The real object in view, however, is to obtain as exactly as possible the average composition of the canes as they are delivered to the factories for commercial purposes, and this is what has been accomplished by this investigation.

The data show that the manufacture of sugar on a commercial scale from such canes would probably not prove successful. The content of sucrose is quite as high as that of the canes used for sugar making in Louisiana, but the manufacturing season is shorter. Such canes worked by the best modern appliances would yield about 180 pounds of merchantable sugar per ton, of which from 140 to 150 pounds would be high grade, first sugars, and the rest low grade, second and third sugars. For sirup making, however, these canes are of excellent quality. The high quantity of reducing sugar, which interferes with sugar manufacture, is no bar to sirup manufacture; on the contrary it is an advantage, as the reducing sugar is quite as sweet and palatable as sucrose and has a much lower coefficient of crystallization. Hence its presence in the product, while impairing neither its appearance nor its taste, improves the selling qualities of the sirup by diminishing the tendency to crystallization.

O

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY—BULLETIN NO. 76.
H. W. WILEY, Chief of Bureau.

INSECTICIDE STUDIES.

I. PYRETHRUM POWDERS CONTAINING POISONOUS METALS.
II. A COMPILATION OF ANALYSES OF INSECTICIDES AND FUNGICIDES.
III. STATE LAWS GOVERNING THE COMPOSITION AND SALE OF INSECTICIDES.

BY

J. K. HAYWOOD,
Chief, Insecticide and Agricultural Water Laboratory.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1903.

LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., March 15, 1903.

Sir: I have the honor to transmit for your approval three papers on insecticides prepared in the Insecticide and Agricultural Water Laboratory of this Bureau, and to recommend their publication as Bulletin No. 76 of the Bureau of Chemistry.

Respectfully,

H. W. Wiley,
Chief.

Hon. James Wilson,
Secretary of Agriculture.

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INSECTICIDE STUDIES.

I. PYRETHRUM POWDERS CONTAINING POISONOUS METALS.

INTRODUCTION.

In the course of a recent examination of a large number of miscellaneous insecticides it was observed that a number of pyrethrum powders contained lead chromate. These powders are sold under such names as Persian Insect Powder, Dalmatian Insect Powder, Buhach, and Pyrethrum, and are colored yellow because there seems to be a popular demand for this grade of goods, the public apparently believing that the preparation must be bright yellow in order to kill insects. As a matter of fact pure pyrethrum, which is composed of the ground flower heads of *Pyrethrum roseum* and *Pyrethrum cinerariæfolium*, usually possesses a light yellow color, but never the bright, intense yellow color given to it by lead chromate.

As lead chromate is known to be poisonous, it would appear that it is a very reprehensible practice to add it to pyrethrum powders, since they are often blown about living rooms with a bellows to kill insects, and are consequently inhaled by human beings. Besides this, pyrethrum is always advertised to be harmless to human beings, which it is when pure, but ceases to be so when it has been mixed with lead chromate or other yellow colored chromates. In view of the above facts it was deemed advisable to examine the pyrethrum powders sold upon the American market to discover which of them contain yellow chromates and at the same time to cite cases of poisoning by chromates to substantiate the claim that any of the yellow chromates that might be used for this purpose, and especially lead chromate, are poisonous to human beings. The facts which have been collected on these points are embodied in the following report:

CASES OF POISONING BY LEAD AND OTHER CHROMATES.

Since it is not generally known that many pyrethrum powders contain lead chromate, it doubtless often happens that physicians are much puzzled as to the cause of the illness of persons who have been exposed to the action of such powders. In order, therefore, that physicians and others may recognize the symptoms of chromate poisoning caused by this class of goods, the following account is given of cases of chromate poisoning found in medical records:

LEAD POISONING CAUSED BY THE MANUFACTURE OF FUSES FOR LIGHTING PURPOSES.[a]

On February 22, 1875, a woman of about 46 years of age came under my care at the hospital. She complained of pains in the stomach and a general indisposition or breaking down. For a number of days she had suffered with severe gripes. Her stomach was not swollen, but her skin was very sensitive and palpation caused a contraction of the abdominal muscles. There was, however, no tumor in the abdominal cavity. She had suffered for about fifteen days with obstinate constipation that could be relieved only by an injection; her liver was small and not painful; her tongue white, appetite nearly entirely lacking, and breath foul. There was nausea and a distaste for food, and one could observe the existence of a grayish-black slate-colored border on the edges of her teeth next to the gums, which had all the characteristics of lead bands. That symptom, added to the gripes and to cramps in the calves of the legs during the night, caused me to diagnose the case as lead poisoning. This woman was under my care until the 16th of March, at which time she began to convalesce, although at that time she was still anæmic and feeble; but there was unquestionable improvement.

Being well convinced that my patient was suffering with lead poisoning, I thought I ought to find the cause. I found that she had worked for five years in a well-aired factory employing about 30 people, where they were engaged in the manufacture of fuses which were to be used by smokers to light their pipes and cigars. Her work differed from that of her fellow-workers in that she was employed in winding the cotton that was to be used for the manufacture of fuses, while they wove the threads of cotton. She was thus more exposed than her companions to the emanating dust, especially as the cold weather had obliged them to close the windows of the shop. These fuses were about as large around as a pencil or the little finger and of a yellowish orange color, indicating that they were colored with lead chromate. A chemical analysis showed the presence of this compound.

At the shop where these fuses were manufactured I found that those who were engaged in weaving them did not show any appreciable symptoms of lead poisoning, while those who were employed to shake out and wind the cotton all had fairly well developed cases. The merchant at Paris who manufactured these fuses said that they were originated in Germany, but were now exported largely, especially to America.

FATAL POISONING BY EATING CONFECTIONERY COLORED WITH LEAD CHROMATE.[b]

On March 14, 187–, between 9 and 11 a. m., the two sons of Mr. H., aged respectively 3½ and 1¾ years, ate several small ornaments representing bees that had been placed on a cake. In the afternoon of the same day they became very ill and were attacked with severe vomiting and great prostration. The vomited mass was at first yellow, and was thrown up with great pain. Vomiting continued until 11 p. m., after which it became infrequent. When the physician arrived, at 6 p. m., they were in bed and suffering extremely. They had reddened faces, complained of great thirst, were languid, had no dysentery, although they complained of slight pains.

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[a] Annales d'hygiène, 2d series, No. 44, 1875.

[b] Vierteljahresschrift für gerichtl. Medizin, 1874, **20:** 60.

It was easy to see that they had been poisoned, and since in the morning they had eaten the same food as the rest of the household, who remained in perfect health, probably the poisoning was due to the above-mentioned bees, seven of which had been given to the children to play with, while only one could be found. They finally confessed that they had eaten the remaining six, and the nurse had seen them do it. The nurse thinking it might be harmful, had taken a softened "bee" from the younger child and wiped out his mouth.

One of the "bees," of which more were found at the confectioner's who had made the cake, was examined by a chemist and found to consist of gum and lead chromate. On the following day both patients had the same red and heated faces; they showed no interest, and on being questioned gave tokens of displeasure. They did not complain of local pains, evidently because their senses were benumbed. The younger had some diarrhea and toward noon convulsions, from which his face was livid. The convulsions became more frequent toward evening, and at 9 o'clock on the morning of the following day, March 16, the younger boy died.

The older child still had a red, heated face and displayed no interest, being almost unconscious. The skin upon his breast and abdomen was strikingly red. The evening temperature was 39.5° in the armpits. On the 17th the pulse was irregular and the temperature in the evening 39.6°. Drinking was extremely painful, in spite of great thirst. On the 18th a foul smell came from the mouth, redness was still present, accompanied by stupidity and painful swallowing; temperature in the evening was 39.2°. On the 19th a collapse took place, a very foul smell came from the mouth, and swallowing was almost impossible. The senses were very much benumbed, and death followed at 11 a. m. The result of the post-mortem may be summed up in the words of the physician as follows: "The death of the children is the direct result of the destruction which has befallen the digestive canal from the throat to the duodenum. Concerning the cause of this destruction and concerning some further conditions we will not express an explicit opinion." The liver, stomach and contents, and urine were then subjected to a chemical examination. This gave negative results, except that in the liver of both corpses traces of copper were found, a discovery of no toxological interest. The absence of all poisonous metals in all other parts of the corpses was shown.

The "bees" were also examined, and 0.27 gram of gum and 0.0042 gram of neutral lead chromate were found in one that was 13 mm long and 5 mm thick. The powder used by the confectioner was also analyzed and found to contain lead chromate. The illness of both children, occurring at the same time and showing itself by severe vomiting, must be traced back to their eating some poisonous food; and since it was known that they both ate the "bees" which contained lead chromate and that they vomited up yellow vomit, the eating of the metallic salt must be considered as the cause of the deaths.

It is not remarkable that no lead chromate was found in the alimentary tract, since the children vomited a great deal and thus threw up all of the poison.

FATAL POISONING BY INHALING DUST FROM YARN COLORED WITH LEAD CHROMATE.[a]

On December 29, 1876, a weaver, B., at E., received a package of yellow threads, colored with lead chromate, which were to be used for the manufacture of curtains. B. and his mate, G., worked at one spool and Mrs. B. and a girl, L., at the other. They worked from 7 a. m. to 10 and 11 p. m. While they were working the threads shed powder, according to B.'s statement, so that their faces and hair became entirely yellow. Their spittle appeared yellow, and the powder had a bitter taste. According to Mrs. B. the powder had a sweetish-bitter taste. The male helper sickened after working upon the threads about eight days; he had a headache, with a ringing in the ears, pains in his breast and stomach, and lost his appetite. He had a desire to vomit and was constipated. This continued for six weeks, until he ceased working and went to the doctor. Mrs. B. during the meantime became ill; she complained of loss of appetite and pains in the chest. B. on the last day of the third week of the work was attacked with pains in the stomach. Then appeared nausea, languor, and sleeplessness. Eight or ten days later he was obliged to stop work and send for the doctor on account of pains in the bowels and costiveness. The girl helper was also ill, having diarrhea, loss of appetite, and pains in the breast.

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[a] Vierteljahresschrift für gerichtl. Medizin, 1877, **27:** 29 (No. 1).

Dr. J. stated the symptoms of B. and the helpers in the following terms: Yellow-coated tongue and yellow expectoration, entire loss of appetite, nausea, temporary vomiting, and pains in the region of the navel, accompanied by obstinate constipation. The excrement was also colored yellow, and all the patients were very weak. The doctor stated that these symptoms were all caused by breathing yellow dust from the yarn and indicated plainly lead-chromate poisoning.

Although all of the above-mentioned persons finally recovered, B. lost a 9-weeks-old baby, whose death could be ascribed only to the breathing or swallowing of the yarn dust. When the B.'s noticed how much dust was flying about they sought to protect their baby by spreading a white woolen cloth of medium thickness over his face. This cloth was said to have been yellow with the dust all the time, as well as the bed on which the child was lying. The child's crib stood midway between the two B.'s stools and at night the mother took the child to bed with her. It was healthy and lively, so that continued poor feeding could not have caused the bad condition of the stomach (jelly-like softening and perforation) observed at the post-mortem. Suddenly, six or eight days before its death, which occurred on February 24, the child was taken sick. Its face became pale and its body hot. It had a couple of loose, yellow actions each day, which were accompanied by restlessness and frequent screaming. The skin upon the breast and stomach became red. In the beginning the baby drank, but refused to eat; later it drank with trouble, and on the day it died it swallowed with difficulty. The lips were dry, the breathing short, and death followed slowly. It is to be remarked that the child, after remaining healthy for six weeks in spite of the surrounding yarn dust, was suddenly taken sick six or eight days before death and grew worse at a time when the yellow yarn was not being worked upon. Since the child remained covered with the cloth up to the time of its death, it appears likely that the dust gradually worked its way through the cloth and sifted down so that the child always had to breathe this impure air until its death.

A chemical examination was made of the following organs of the child and the articles that were used at the the time of his death: Contents of the nose, contents of the stomach, urine and gall taken from the urine and gall bladders, organs of the stomach and breast, the hair of the head, shirt slip, the child's nipple, and a portion of the yellow yarn. The brain would have been examined, but the vessel containing it was lost. The yarn contained 11.83 per cent lead chromate. Lead chromate was found in the hair of the head, and the little slip contained 65 mg lightly strewn over it. The organs of the neck, chest, and gullet contained 36 mg of lead chromate. The other fluids of the body and organs, the shirt, and nipple did not show the presence of any lead chromate.

It will thus be seen that numbers of authentic cases of poisoning, by either breathing or swallowing lead chromate, are on record. Following will be found several typical cases of poisoning by alkaline chromates and bichromates selected from the large number that have been observed by Messrs. Delpech and Hillaire:[a]

MEMOIRS CONCERNING DANGERS TO WHICH WORKMEN IN CHROMATE FACTORIES ARE EXPOSED.

OBSERVATION I.—*Workmen employed for a long time at the grinder (pilerie) and formerly at the kettles (chaudières)—Nasal partition perforated by the dust.*

V., aged 50, has worked for twelve years (1849) in a chromate manufacturing plant. He was employed from the beginning at the grinder and sifter, and is thus constantly enveloped in an atmosphere charged with fine chrome-iron powder. He says that he feels no indisposition. He wipes his nose and spits blackish matter, but neither coughs nor has any smothered feeling; his health appears to be good; he has no disease of the lungs; he does not believe there is anything the matter with his nose; however, his sense of smell was perfect when he entered the factory, and he has now completely lost it. The nasal passages were examined, and a perforation of the cartilage was observed. He says that since entering the factory he has sometimes worked at the kettles. At the commencement of this labor he was attacked with headache and swelling of the nose accompanied by an abundant discharge from the nostrils. His eyes did not smart. These symptoms lasted about fifteen days, after which he regained the most perfect health without even experiencing again the symptoms enumerated, although employed at the same work.

This workman has since (October, 1864) died after a few days' illness of inflammation of the chest, without having previously shown any prolonged symptoms of suffering with the respiratory organs.

OBSERVATION III.—*Workman employed at the kettles for about four weeks—Inflammation of the mucous membrane of the nose—Sore upon the right thumb—A nasal perforation—Ulceration of both of the external nasal walls.*

E., aged 28, who had a good constitution, had been employed for five weeks in a potassium bichromate manufacturing plant when he was examined by us. He was employed about eight days inside the works carrying the bichromate and for three weeks at the kettles. From the first day he was attacked by prolonged and intense sneezing. After four days he suffered greatly with intense prickling, abundant running of the nose, an incessant desire to blow his nose, and the passage of red membraneous shreds. He had no watering of the eyes, or at most it was slight. The most severe symptoms lasted from ten to twelve days. He had nearly recovered when he stopped working at the kettle, and has since completely recovered. This workman still has his sense of smell. He was afflicted by a sore on his right hand, caused by penetration of particles of bichromate in a slight excoriation, and a slight ulceration still remains. One can also note brown spots, caused by sores of the same nature, upon the side of his face and on the ring finger.

E. has never suffered with his feet. He has no eruption either on the skin of his body or genital organs. His eyelids are not excoriated, and he has neither vomited, suffered with diarrhea, nor a cough. The nasal partition is perforated at the usual place.

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[a] Annales d'Hygiène Publique et de Medicine Legale, 1869, and 1876, 2d series.

OBSERVATION VI.—*Workman, a coppersmith unaccustomed to the manufacture of chromate, was exposed to the dust resulting from the repairing of the kettles—Inflammation of the mucous membrane of the nose and nasal perforation.*

F., a coppersmith and a stranger to the chromate factory, came into the factory for a short time to repair the kettles. This repairing required a readjusting of the sides by hitting with a hammer, and these blows caused the crust of bichromate that adheres in the slightest irregularities to fly about in the form of a dust. He was attacked quickly by inflammation of the mucous membrane of the nose and lost his nasal partition in eight days.

OBSERVATION XI.—*Superintendent, not employed at any special work—Chronic irritation, swelling of the membrane at the usual place, but no perforation.*

O., aged 20 (December, 1863), has been employed for four years in the chromate works. He has never worked at the kettles or the calciner. Charged with general surveillance, he merely walks through the works; in exceptional cases, however, he helps solder the lead crystallizing vats that are in need of repairs. He has consequently to a certain extent been subjected to the action of bichromate. At the time we examined him he had felt a tingling of the nose and had frequent fits of sneezing, but his nasal membrane had not been badly inflamed. His nasal partition is not perforated, but at the usual point he has a slight swelling and reddening of the membrane. His general health is good.

OBSERVATION XVIII.—*Fireman exposed to chromate dust—Perforation of the nasal cartilage, ulcerated sores on the right hand.*

D., aged 28, has been employed at the works for two years, and for the last ten months at the manufacture of potassium chromate. He is employed as a fireman, but often helps his fellow-workmen, and so during much of the time spent in the factory he is exposed to chromate dust. He had always enjoyed the best of health. He had never had syphilitic symptoms, but during his infancy the glands at the side of his neck were swollen for a long time. His general condition is good. He had worked at the manufacture of potassium chromate for scarcely fifteen days when he was taken with nose bleeding, sneezing, pain in the nose, and watering of the eyes. He sometimes experienced a little uneasiness in breathing. It was fifteen or twenty days after he had ceased to work at the preparation of potassium chromate that he examined his nose and found a small perforation. The nose preserves its normal form. There is an ulcer on the nasal cartilage in the right nostril, and at the edges of this the mucous membrane is very much inflamed. In the left nostril is an opening corresponding to this ulceration. His sense of smell is preserved; neither the throat, palate, nor roof of the mouth have been attacked, and he has no cough. This workman has hard, cartilaginous, livid scars upon his thumb and his right hand. On the left index finger there exists a sore which is much swollen and exudes a sero-purulent liquid. This workman has never had any swelling or ulceration of his feet.

There are many other observations cited in the above article, but enough have been given to show that persons who are obliged to breathe air charged with the dust from chromates or bichromates are nearly always affected more or less seriously.

ANALYSES OF PYRETHRUM POWDERS.

COLLECTION OF SAMPLES.

In order that samples of pyrethrum, representative of all grades, might be obtained, a circular letter was forwarded to the chemist of each of the experiment stations, with the request that they purchase for us, upon the open market, three or four samples of those products sold under the name of either Pyrethrum, Buhach, Persian Insect Powder, or Dalmatian Insect Powder. They were also asked to give data as to the price per two-ounce sample, the name and address of the retail dealer, and the name and address of the person who originally prepared the sample. In answer to these letters 105 samples were received, all of which were subjected to chemical examination. It is believed that these samples represent fairly well the present state of the American market.

Credit is due to the assistants in the laboratory, Bernard H. Smith and Harry W. Houghton, who aided in performing the analytical work.

METHODS AND RESULTS OF ANALYSIS.

Since lead, potassium, and barium chromate have all a bright yellow color, and any of them might be used to color the pyrethrum powders, tests for all of these substances were made in the samples under examination according to the following methods:

Qualitative test for lead.—Ash about 2 grams of the sample in porcelain and treat with a little water and about 20 cc of concentrated hydrochloric acid in a porcelain dish. Place on the steam bath and steam for about one-half an hour or until all the lead is in solution and the chromium reduced and in solution. Filter after dilution. Add sodium carbonate until the solution is alkaline and then add acetic acid until the solution is just acid. Filter and add a solution of potassium chromate. A yellow precipitate or cloudiness indicates lead.

Qualitative test for barium.—Ash about 2 grams of the sample in porcelain and treat with hydrochloric acid as directed for lead. Evaporate to dryness and take up with about 0.8 cc of hydrochloric acid. Filter and pass hydrogen sulphid through the filtrate. Filter off any sulphids that may precipitate and test for barium in the filtrate with a few drops of dilute sulphuric acid.

Qualitative test for chromium.—Ash about 2 grams of the sample in porcelain. Treat this with about four times its weight of a mixture of equal parts of sodium carbonate and potassium nitrate, and fuse. Dissolve in water on the steam bath and filter. Make slightly acid with acetic acid and test with lead acetate for the presence of chromium.

Testing the powders by the above methods showed that whenever chromium was present lead was also present, these two evidently being combined as lead chromate. A quantitative estimation was also made of the chromium present according to the following method of analysis and the chromium was calculated to lead chromate.

TABLE I.—*Description of samples, source, and price.*

Serial No.	Name.	Retailer.	Manufacturer or wholesaler.	Price per 2 ounces.
266	Persian Insect Powder	G. B. Evans, Philadelphia, Pa	G. B. Evans, Philadelphia, Pa	$0.10
267	Dalmatian Insect Powder	Llewellyn, Philadelphia, Pa	Llewellyn, Philadelphia, Pa	.25
268	Insect Powder	Stoever, Philadelphia, Pa		.10
269	Pyrethrum	Menges, Madison, Wis	Allaire, Woodward & Co., Peoria, Ill	.10
270	do	Henry Lewis, Madison, Wis	Fuller & Fuller, Chicago, Ill	.05
271	Dalmatian Insect Powder	Hollister's Pharmacy, Madison, Wis	Allaire, Woodward & Co., Peoria, Ill	.10
272	Insect Powder	F. L. Gardner, Lansing, Mich	Parke, Davis & Co., Detroit, Mich	
273	Dalmatian Insect Powder	Robinson Drug Co., Lansing, Mich	do	
274	Spalato Insect Powder	Bauer's Drug Co., Lansing, Mich	Lehn & Fink, New York, N. Y	
275	Dalmatian Insect Powder	M. A. Dickinson, Amherst, Mass	Archibald & Lewis, New York, N. Y	.10
276	Persian Insect Powder	W. S. Wescott, Amherst, Mass	Hoyt Medicine Co., New York, N. Y	.08
277	Dalmatian Insect Powder	Holland & Gallord, Amherst, Mass	Lee & Osgood, Norwich, Conn	.10
278	do	H. Adams, Amherst, Mass	E. L. Patch Co. (wholesaler), Boston, Mass	.10
279	Insect Powder	Charles Deuel, Amherst, Mass	Schieffelin & Co., (wholesaler) New York, N. Y	.15
280	do	Rector's Pharmacy, Lincoln, Nebr	Allaire, Woodward & Co., Peoria, Ill	.20
281	do	Harley Drug Co., Lincoln, Nebr	do	.05
282	Buhach	H. W. Brown, Lincoln, Nebr	Buhach Manufacturing Co., Stockton, Cal	.25
283	Pyrethrum	do	Richardson Drug Co., Omaha, Nebr	.13
312	Insect Powder	C. Powells, Stillwater, Okla		.10
313	Pyrethrum	W. H. Hand, Stillwater, Okla	Parke, Davis & Co., Detroit, Mich	.10
314	Insect Powder	Carl Engel, Manhattan, Kans		.05
315	Pyrethrum	Koller & Masters, Manhattan, Kans		.10
316	do	G. B. Harrot, Manhattan, Kans	Faxon, Horton & Gallagher, Kansas City, Mo	.15
317	Dalmatian Insect Powder	Orr Drug Co., Athens, Ga	Gilpin, Langdon & Co., Baltimore, Md	.10
318	do	Palmer & Kinnebum, Athens, Ga		.10
319	Persian Insect Powder	H. O. Meck, State College, Pa	Smith, Kline & French, Philadelphia, Pa	.10
323	Buhach	George Martin, Tucson, Ariz	Buhach Manufacturing Co., Stockton, Cal	.15
324	Pyrethrum	B. L. Read, Bryan, Tex	Parke, Davis & Co., Detroit, Mich	.05
325	do		Eimer & Amend, New York, N. Y	.05
326	do	M. H. James, Bryan, Tex	Nickells Stone-Chemical Co., New Orleans, La	.05
327	Persian Insect Powder	The Van Deusen Pharmacy, New Brunswick, N. J.		.10
328	do	William Rust & Sons, New Brunswick, N. J	William Rust & Sons, New Brunswick, N. J	.10
329	do	Skillman & Van Pelt, New Brunswick, N. J		.10
330	Insect Powder	L. Christianson, Fargo, N. Dak		.05
331	do	R. H. Finney, Fargo, N. Dak	Parke, Davis & Co., Detroit, Mich	.10
332	do	Font & Portfield, Fargo, N. Dak	Noyer Bros. & Cutter, Fargo, N. Dak	.10
333	do	B. F. Mackaff, Moorhead, Minn	Lyman Eliet Drug Co., Minneapolis, Minn	.10
334	do	Waldorf Pharmacy, Fargo, N. Dak	Parke, Davis & Co., Detroit, Mich	.05
335	do	G. C. Sadin, Fargo, N. Dak		.10
336	Pyrethrum	J. E. Knight, Exeter, N. H		.10
337	do	A. S. Wethereff, Exeter, N. H		.10
338	do	J. C. Vickery, Dover, N. H		.10
339	Persian Insect Powder	H. E. Glick, Lafayette, Ind		.10
340	Pyrethrum	Varoter-Boggs Drug Co., Lafayette, Ind	Allaire, Woodward & Co., Peoria, Ill	.05

341	....do	Hogan-Johnson Drug Co., Lafayette, Ind	Stollman & Fulton, New York, N. Y	.05
384	Dalmatian Insect Powder	D. B. Sloan, Clemson College, S. C	F. H. Leggett, New York, N. Y	.15
385	....do	E. G. Evans, jr., & Co., Pendleton, S. C	Parke, Davis & Co., Detroit, Mich	.10
386	Persian Insect Powder	R. D. Stearns, Burlington, Vt	Schieffelin & Co., New York, N. Y	.10
387	....do	C. A. Barber, Burlington, Vt	J. E. Gary & Co., Boston, Mass	.10
388	....do	G. A. Churchhill, Burlington, Vt	McKesson & Robbins, New York, N. Y	.10
389	....do	G. E. Perkins, Burlington, Vt	Burlington Drug Co., Burlington, Vt	.10
390	....do	J. E. Bellrobs, Burlington, Vt	Polk & Colder, Troy, N. Y	.10
391	....do	Crystal Pharmacy, Burlington, Vt	Parke, Davis & Co., Detroit, Mich	.10
392	Insect Powder	Proctor & O'Hail, Wooster, Ohio		
393	....do	Zimmerman & Co., Wooster, Ohio		
394	....do	Sauback & Boyd, Wooster, Ohio	Allaire, Woodward & Co., Peoria, Ill	
395	Buhach	Graham & Wortham, Corvallis, Oreg	Buhach Manufacturing Co., Stockton, Cal	.05
396	Persian Insect Powder	Graham & Wells, Corvallis, Oreg		
397	....do	Allen & Woodward, Corvallis, Oreg	Blumaleer, Frank & Co., Portland, Oreg	
401	Dalmatian Insect Powder		Lehn & Fink, New York, N. Y	.05
402	Pyrethrum	Owens & Owens, Wytheville, Va	Parke, Davis & Co., Detroit, Mich	.10
403	Persian Insect Powder	Ellett's Drug Store, Blacksburg, Va	....do	.05
404	Black Flag Insect Powder	Owens & Owens, Wytheville, Va	Nathan Howe, Boston, Mass	.15
419	Pyrethrum	White & Burdick, Ithaca, N. Y	Leggett Bros., New York, N. Y	
420	....do	Brooks's Drug Store, Ithaca, N. Y	B. Ritchey & Co., New York, N. Y	
421	Insect Powder	P. Simmon, Danbury, Conn		.10
422	Persian Insect Powder	A. Dutschbeyer, Hartford, Conn		.10
423	Insect Powder	E. D. Weed, Norwalk, Conn		.10
424	....do	J. M. Cotter & Co., New Britain, Conn		.10
425	....do	Park Drug Co., Hartford, Conn		.05
426	....do	C. H. Belle, Hartford, Conn		.10
427	Dalmatian Insect Powder	C. L. Finch, Norwalk, Conn		.10
428	Insect Powder	G. S. Coburn, New Haven, Conn		.20
429	....do	G. R. Steele, Thompsonville, Conn		.10
430	....do	Hull's Drug Store, New Haven, Conn		.10
431	....do	Hartigan Pharmacy, Bridgeport, Conn		.05
432	....do	Waterbury Drug Co., Waterbury, Conn		.10
433	....do	J. A. McMillan & Co., Knoxville, Tenn		.05
434	....do	W. W. Hall, Knoxville, Tenn	Sanford, Chamberlain & Albers, Knoxville, Tenn	.10
435	....do	H. Kuhlman, Knoxville, Tenn	McKesson & Robbins, New York, N. Y	.10
492	Dalmatian Insect Powder	The Laramie Drug Co., Laramie, Wyo		.20
493	Buhach	....do	Buhach Manufacturing Co., Stockton, Cal	.20
494	Pyrethrum	Ticknor & Jagger, St. Paul, Minn		.20
495	Insect Powder	M. L. Payant, Faribault, Minn		.10
496	....do	Dillin Drug Co., Minneapolis, Minn	Allaire, Woodward & Co., Peoria, Ill	
509	Pyrethrum	J. T. Tidball, Brookings, S. Dak	Fuller & Fuller, Chicago, Ill	.10
510	Persian Insect Powder	....do	Parke, Davis & Co., Detroit, Mich	.10
511	Pyrethrum	Irwin Pharmacy, Brookings, S. Dak	....do	.10
512	Dalmatian Insect Powder	Johnson-Platt Drug Co., Salt Lake City, Utah		.10
513	Buhach	Willis-Horne Drug Co., Salt Lake City, Utah	Buhach Manufacturing Co., Stockton, Cal	.10
591	....do	J. J. Argenti, San Francisco, Cal	Allaire, Woodward & Co., Peoria, Ill	.10
592	....do	Wm. Scarby, San Francisco, Cal	California Insect Powder Co., Stockton, Cal	.10
593	Persian Insect Powder	Langley & Michaels, San Francisco, Cal	Thursden & Bradish, New York, N. Y	.10
594	Buhach	....do	Buhach Manufacturing Co., Stockton, Cal	.10
642	Pyrethrum	C. T. Carr & Bro., Hyattsville, Md		.20
647	Insect Powder	Thomson & Hogan, Christiansburg, Va	Parke, Davis & Co., Detroit, Mich	

TABLE I.—*Description of samples, source, and price*—Continued.

Serial No.	Name.	Retailer.	Manufacturer or wholesaler.	Price per 2 ounces.
648	Insect powder	R. S. Carson, East Bradford, Va	Parks, Davis & Co., Detroit, Mich	
649	Persian Insect Powder	G. W. Lyle, Bradford, Va	do	$0.24
693	Buhach	C. L. McCartor, Moscow, Idaho	Buhach Manufacturing Co., Stockton, Cal	.15
694	Insect Powder	L. H. Torsen, Moscow, Idaho	Parke, Davis & Co., Detroit, Mich	.10
695	Persian Insect Powder	A. K. Greenman, Kingston, R. I		.10
696	do		Brownell & Field, Providence, R. I	.10
697	Insect Powder		Oliver Johnson & Co., Providence, R. I	.10
698	Castillian Insect Powder		Austin, Nichols & Co., New York, N. Y	.15
[a] 19530	Persian Insect Powder	Z. D. Gilman, Washington, D. C	Z. D. Gilman, Washington, D. C	.10

[a] General index number.

Method for determining chromic acid.—Weigh out from 2 to 4 grams of the sample. Ash in a porcelain dish and allow to cool. Treat with a little water and a large amount of concentrated hydrochloric acid and evaporate to dryness. Treat again with concentrated hydrochloric acid and evaporate to dryness.

Take up the residue with about 0.8 cc of concentrated hydrochloric acid with the aid of heat. Filter and pass hydrogen sulphid through the solution until all lead is precipitated. Filter and evaporate the filtrate to dryness. Take up the residue with a little water and 1 cc of hydrochloric acid with the aid of heat. Precipitate out the chromium (with iron, aluminum, and some phosphoric acid) by means of ammonia, taking care not to add any excess of ammonia. Filter and wash with hot water. Dissolve the precipitate on the filter by means of hot dilute hydrochloric acid and evaporate to dryness. Take up with a little hot water and 6 to 8 drops of concentrated hydrochloric acid, heating for a short time if necessary. Transfer to a 50 cc or 100 cc flask and make up to the mark. Now prepare an alkaline permanganate solution in the following manner: Make an approximately N/10 solution of potassium permanganate and standardize it against ammonium ferrous sulphate. Measure out exactly a liter of this solution and add a few grams of sodium carbonate and 1 gram of sodium hydroxid and make the entire volume up to 2 liters. This is the standard solution required.

A convenient number of cubic centimeters of this standard is measured into a beaker and about 50 cc of distilled water, accompanied by a little sodium carbonate, is added. The solution of chromium previously prepared is then run in from a burette, while the contents of the beaker are boiling, until the pink color is dissipated and a pure yellow color results. Knowing the strength of the permanganate and the number of cubic centimeters of the chromium solution used, one can calculate the percentage of chromium in the original powder according to the following equation:

$$CrCl_3 + KMnO_4 + 4\ KOH = K_2CrO_4 + MnO_2 + 3\ KCl + 2H_2O$$

In Table II are found the results obtained upon the samples of pyrethrum powder examined:

TABLE II.—*Examination of pyrethrum powders for coloring matter.*[a]

Serial No.	Name.	Chromium.	Lead.	Chromium trioxid (CrO_3).	Lead chromate ($PbCrO_4$).
				Per cent.	*Per cent.*
266	Persian Insect Powder	Absent	Absent		
267	Dalmatian Insect Powder	Present	Present	0.099	0.32
268	Insect Powder	do	do	.071	.23
269	Pyrethrum	Absent	Absent		
270	do	do	do		
271	Dalmatian Insect Powder	do	do		
272	Insect Powder	do	do		

[a] Examination for barium gave negative results for all samples.

TABLE II.—*Examination of pyrethrum powders for coloring matter*—Continued.

Serial No.	Name.	Chromium.	Lead.	Chromium trioxid (CrO_3).	Lead chromate ($PbCrO_4$).
				Per cent.	*Per cent.*
[a]273	Dalmatian Insect Powder	Present	Present	.068	.22
274	Spalato Insect Powder	Absent	Absent		
275	Dalmatian Insect Powder	Present	Present	.130	.42
276	Persian Insect Powder	Absent	Absent		
277	Dalmatian Insect Powder	Present	Present	.068	.22
278	do	Absent	Absent		
279	Insect Powder	do	do		
280	do	do	do		
281	do	do	do		
282	Buhach	do	do		
283	Pyrethrum	do	do		
312	Insect Powder	do	do		
313	Pyrethrum	do	do		
314	Insect Powder	do	do		
315	Pyrethrum	Present	Present	.124	.40
316	do	do	do	.102	.33
317	Dalmatian Insect Powder	Absent	Absent		
318	do	do	do		
[a]319	Persian Insect Powder	Present	Present	.186	.60
323	Buhach	Absent	Absent		
324	Pyrethrum	do	do		
325	do	do	do		
326	do	Present	Present	.251	.81
327	Persian Insect Powder	do	do	.365	1.18
328	do	Absent	Absent		
329	do	Present	Present	.421	1.36
330	Insect Powder	Absent	Absent		
331	do	do	do		
332	do	do	do		
333	do	do	do		
334	do	do	do		
335	do	do	do		
336	Pyrethrum	do	do		
337	do	do	do		
338	do	do	do		
339	Persian Insect Powder	do	do		
[a]340	Pyrethrum	Present	Present	.090	.29
341	do	Absent	Absent		
384	Dalmatian Insect Powder	do	do		
385	do	do	do		
[a]386	Persian Insect Powder	Present	Present	.043	.14
387	Insect Powder	Absent	Absent		
388	Persian Insect Powder	do	do		
389	do	do	do		
390	do	do	do		
391	do	do	do		
392	Insect Powder	do	do		
393	do	do	do		
394	do	do	do		
395	Buhach	do	do		
396	Persian Insect Powder	do	do		
397	do	do	do		
401	Dalmatian Insect Powder	do	do		
402	Pyrethrum	do	do		
403	Persian Insect Powder	do	do		
404	Black Flag Insect Powder	do	do		
419	Pyrethrum	Present	Present	.037	.12
420	do	do	do	.056	.18
421	Insect Powder	Absent	Absent		
422	Persian Insect Powder	do	do		
423	Insect Powder	Present	Present	.186	.60
424	do	Absent	Absent		
425	do	do	do		
426	do	do	do		
427	Dalmatian Insect Powder	Present	Present	.362	1.17
428	Insect Powder	Absent	Absent		
429	do	do	do		
430	do	do	do		
431	do	do	do		
432	do	do	do		
433	do	do	do		
434	do	do	do		
435	do	do	do		
492	Dalmatian Insect Powder	do	do		
493	Buhach	do	do		
494	Pyrethrum	do	do		
495	Insect Powder	do	do		

[a] See correspondence with manufacturers, p. 20.

TABLE II.—*Examination of pyrethrum powders for coloring matter*—Continued.

Serial No.	Name.	Chromium.	Lead.	Chromium trioxid (CrO_3).	Lead chromate ($PbCrO_4$).
				Per cent.	*Per cent.*
496	Insect powder	Absent	Absent		
509	Pyrethrum	Present	Present	.046	.15
510	Persian Insect Powder	Absent	Absent		
511	Pyrethrum	do	do		
512	Dalmatian Insect Powder	do	do		
513	Buhach	do	do		
591	do	do	do		
592	do	do	do		
593	Persian Insect Powder	do	do		
594	Buhach	do	do		
642	Pyrethrum	do	do		
647	Insect Powder	do	do		
648	do	do	do		
649	Persian Insect Powder	do	do		
693	Buhach	do	do		
694	Insect Powder	do	do		
695	Persian Insect Powder	do	do		
696	do	do	do		
697	Insect Powder	do	do		
[a] 19530	Persian Insect Powder	Present	Present	.455	1.47

[a] General index number.

A glance at this table shows that wherever the powders are colored by a foreign substance lead chromate is the compound used. Nineteen of the 105 samples, or 18 per cent, were colored by lead chromate, the amount of which varied from 0.12 to 1.47 per cent.

Of the adulterated samples, 5 were sold under the name of Dalmatian Insect Powder, 5 under the name of Persian Insect Powder, 7 under the name of pyrethrum, and 2 simply as insect powder. It is also evident from Tables I and II that some firms are sending out two grades of pyrethrum, one containing lead chromate and the other not containing that substance. It is unfortunate that the names of all those who originally prepared the above samples could not be obtained since the powders containing the largest amount of lead chromate lack the name of the manufacturer with surprising regularity.

It seems hardly necessary to call attention to the widespread harm that is undoubtedly caused by powders thus colored. They are sold in all parts of the country and are especially advocated to kill or drive away flies, fleas, cockroaches, and other household pests, and thus they are used principally in the home. Consequently they are breathed by many people for days and weeks at a time. Such powders not only possess the poisonous characteristics of nearly all lead salts, but in addition have the toxic properties of chromic acid. It would appear, therefore, that the influence of lead chromate day by day no doubt occasions many of the mysterious cases of lead poisoning in which it seems impossible for the physician to find the source of the lead taken into the system.

CORRESPONDENCE WITH MANUFACTURERS.

In order that the manufacturers might have an opportunity to comment upon the analyses before they were published, a circular letter was sent to each one whose goods had been examined, giving him the results of our analysis and asking for any comments that he might desire to make. Such parts of the answers as throw any light upon the subject are given, as follows:

Serial No. 340.

* * * You say that the Varoter-Boggs Drug Company, of Lafayette, Ind., furnished you with a sample of our insect powder containing 0.29 per cent of lead chromate. If such is the case, they added the lead chromate themselves, as we do not use it. We do not color our insect powder, but leave that for the customers to do if their trade demands a highly colored powder.

ALLAIRE, WOODWARD & CO.

Comment: It appears that the above sample was probably colored after it left the hands of the manufacturer, since seven other samples of Allaire, Woodward & Co.'s goods (Nos. 269, 271, 280, 281, 394, 496, and 591) contained no lead chromate.

Serial No. 273.

* * * From the manager of our importing department we also receive the positive assurance that the powder entering into your sample No. 273 could not possibly have contained lead chromate as it left the hands of his department. It goes without saying that we never add lead chromate or any other foreign substance to our insect powder and that the adulteration which you report must have been deliberately put into it by some interested person after it passed out of our possession.

PARKE, DAVIS & CO.

Comment: It appears from investigations which have been made that the above sample received the lead chromate after it left the hands of the manufacturer named, if indeed it were manufactured by the firm originally. Fourteen other samples of Parke, Davis & Co.'s goods (Nos. 272, 313, 331, 334, 385, 391, 402, 403, 510, 511, 647, 648, 649, 694) contained no lead chromate.

Serial No. 386.

* * * Messrs. R. D. Stearns & Co., Burlington, Vt., are in error as to the source of the Persian insect powder which they furnished your representative, stating that it came from our firm, and we have written them to that effect and asked for an explanation. We have examined their entire account for the year 1902, and positively state that they did not purchase any insect powder of us, and if they had it would have been found to be perfectly pure, as we do not allow any adulteration of Persian powder sold by us.

SCHIEFFELIN & CO.

Comment: It is probable that the retailer made a mistake in giving the source of his insect powder, since another sample furnished by Schieffelin & Co. (No. 279) contained no lead chromate.

Serial No. 319.

* * * We presume that this analysis is approximately correct, and that therefore there is foreign matter in the insect powder amounting to a little over one-half of 1 per cent. This additional matter is undoubtedly added, not for the purpose of adul-

teration, but for the purpose of brightening the color of the goods. * * * Until recent years it was the custom to sell only colored insect powder, which, of course, required the addition of a considerable percentage of coloring matter. Some years ago, however, we and some others decided that we would endeavor to educate the trade to accept only a strictly pure insect powder, uncolored, and we now supply the colored only, we believe, to one customer, who insists on getting the same. * * * The particular lot of which you sent us the test, however, is not of our own powdering. Last year there was a sudden and very large advance in the price of insect flowers, and we therefore bought in this country the insect powder of a brand that has been held to be one of the most reliable and has had an established reputation for over fifty years. The exceedingly small amount of foreign matter added, amounting to a little over 0.5 per cent, proves conclusively that the object was not to adulterate the powder.

SMITH, KLINE & FRENCH CO.

II. A COMPILATION OF ANALYSES OF INSECTICIDES AND FUNGICIDES.[a]

INTRODUCTION.

In a recent publication[b] of the Bureau of Chemistry analyses of all the insecticides and fungicides that could be obtained on the American market were given, together with such remarks concerning their effectiveness as the results seemed to justify. It was originally intended that all of the analyses which had previously been made by the various experiment stations should be included in that bulletin. By reason, however, of unavoidable delays, two years passed between the time of collecting the samples and the time of completing the analyses. Since results of this kind, to be of the most value, should be published at once, it was thought desirable to reserve the compilation of results obtained in the various States for a future publication. This article therefore includes a compilation of analyses of insecticides and fungicides made by the various experiment stations, together with a few analyses made by the Bureau of Chemistry since the publication of Bulletin 68.

The insecticides are arranged in groups, as in the previous bulletin, with the addition of two or three classes not taken up in that publication, and the omission of a few classes not examined by the experiment stations. They will be considered in the following order: (1) Paris greens; (2) London purples; (3) insecticides and fungicides, other than Paris greens and London purples, that contain arsenic, copper, or both; (4) soaps; (5) lyes; (6) hellebores; (7) mixtures containing borax; (8) tobacco extracts; (9) formalins; (10) petroleums; (11) miscellaneous solid insecticides and fungicides, and (12) miscellaneous liquid insecticides and fungicides.

SOURCES OF ANALYSES AND DESCRIPTION OF SAMPLES.

Table I, on the following pages, is introduced to show the publications from which the analyses were taken and, when possible, the names of the manufacturers of the various compounds.

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[a] No responsibility is assumed by the Department for the correctness of compiled analyses, not made within the Department itself.

[b] U. S. Dept. Agr., Bureau of Chemistry Bul. 68, The Chemical Composition of Insecticides and Fungicides.

TABLE I.—*Source of analysis and description of samples.*

Sample No.	Name.	Source.	Manufacturer of sample.
1	Paris Green	Alabama Experiment Station, Bulletin No. 58 (1894)	
2	do	do	
3	do	do	
4	do	do	
5	do	do	
6	do	do	Eimer & Amend, New York City.
7	do	do	
8	do	California Experiment Station Report (1891), p. 79	
9	do	do	
10	do	do	
11	do	do	
12	do	do	
13	do	California Experiment Station Report (1895), p. 137	
14	do	do	
15	Philadelphia Solid Lye	California Experiment Station, Bulletin No. 52	
16	American Concentrated Lye	do	
17	Double Concentrated 98 percent Caustic Soda	do	Green Bark Alkali Works Co., Lancashire, England.
18	Refined Pearl Ashes	do	Do.
19	Commercial Potash	do	Langley & Michaels, San Francisco, Cal.
20	Canadian Crude Potash	do	
21	Paris Green	California Experiment Station, Bulletin No. 126	In New York City.
22	do	do	
23	do	do	
24	do	do	
25	do	do	
26	do	do	
27	White Arsenoid	do	
28	Pink Arsenoid	do	
29	Green Arsenoid	do	
30	Paragrene	do	
31	Paris Green	Florida Experiment Station, Bulletin No. 14 (July 1, 1891)	
32	Concentrated Lye (Potash)	Idaho Agricultural Experiment Station, Bulletin No. 19 (1899).	B. T. Babbitt.
33	American Lye	do	American Lye Co., Philadelphia.
34	Paris Green (average of 7)	do	
35	Sulphur	do	
36	do	do	
37	Paris Green	Idaho Agricultural Experiment Station, Bulletin No. 25 (1901).	
38	do	do	
39	do	do	
40	do	do	
41	do	do	
42	do	do	
43	do	do	

44	....do	....do	
45	....do	....do	
46	....do	....do	
47	....do	....do	
48	....do	....do	
49	....do	....do	
50	....do	....do	
51	....do	....do	
52	....do	....do	
53	....do	....do	
54	....do	....do	
55	....do	....do	
56	....do	Not published, but kindly forwarded by Illinois Agricultural Experiment Station.	A. B. Ansbacher & Co., New York City.
57	Paris Green (Lion brand, new process)	....do	J. A. Blanchard, New York City.
58	Paragrene	....do	F. L. Lavanburg, New York City.
59	Paris Green	....do	
60	....do	....do	H. K. Mulford Co., Chicago, Ill.
61	....do	....do	A. B. Ansbacher & Co., New York City.
62	....do	....do	Fuller & Fuller, Chicago, Ill.
63	....do	....do	
64	....do	....do	
65	Formalin (average of 5)	Indiana Experiment Station Report (1899)	
66	....do	Indiana Experiment Station Report (1900)	Henry Heil & Co.
67	....do	....do	Hogan & Johnson.
68	....do	....do	Eimer & Amend, New York City.
69	Paris Green (average of 10)	Louisiana Experiment Station, Bulletin No. 1, second series (1890), p. 25.	
70	Paris Green	Louisiana Experiment Station, Bulletin No. 23, second series (1893), pp. 778-782.	Sondheim, Alsberg & Co., New York City.
71	Paris Green (average of 2)	Louisiana Experiment Station, Bulletin No. 39, second series (1895), pp. 1431-1434.	
72	Paris Green (average of 8)	Louisiana Experiment Station, Bulletin No. 45, second series (1896), pp. 80-84.	
73	Pine Tar Insecticide	Louisiana Experiment Station, Bulletin No. 48, second series (1897).	
74	Paris Green (average of 39)	Louisiana Experiment Station, Bulletin No. 49, second series (1897), pp. 198-204.	
75	....do	Louisiana Experiment Station, Bulletin No. 54, second series (1898).	
76	Paris Green (average of 36)	Louisiana Experiment Station, Bulletin No. 58, second series (1899).	
77	Paris Green (average of 119)	Louisiana Experiment Station, Bulletin No. 63, second series (1900).	
78	Paris Green (average of 79)	....do	
79	Paris Green (average of 35)	....do	
80	Paris Green (average of 19)	....do	
81	Paris Green	Hatch Experiment Station, Massachusetts, Bulletin No. 38 (1896).	
82	....do	....do	
83	....do	....do	
84	....do	....do	

TABLE I.—*Source of analysis and description of samples*—Continued.

Sample No.	Name.	Source.	Manufacturer of sample.
85	Paris Green (Lion brand, new process)	Hatch Experiment Station, Massachusetts, Bulletin No. 38 (1896).	James Blanchard, New York City.
86	....do	....do	Do.
87	....do	....do	Do.
88	....do	....do	Do.
89	Pink Arsenoid	Hatch Experiment Station, Massachusetts, Bulletin No. 74 (1901).	Adler Color and Chemical Works, New York City.
90	Green Arsenoid	....do	Do.
91	White Arsenoid	....do	Do.
92	Laurel Green	....do	Nichols Chemical Co., Syracuse, N. Y.
93	Bug Death	....do	Danforth Chemical Co., Leominster, Mass.
94	Paris Green	....do	I. Pfeiffer, New York City.
95	....do	....do	A. B. Ansbacher, New York City.
96	....do	....do	Alsberg & Pfeiffer, New York City.
97	....do	....do	Hampden Paint and Chemical Co., Springfield, Mass.
98	....do	....do	Cawley Clark & Co., Newark, N. J.
99	Paris Green (Lion brand)	....do	J. A. Blanchard, New York City.
100	....do	....do	Do.
101	Paris Green	....do	A. B. Ansbacher, New York City.
102	....do	....do	Leggett & Bro., New York City.
103	....do	....do	E. & F. King Company, Boston, Mass.
104	....do	....do	C. T. Reynolds & Co., New York City.
105	Paris Green (Lion brand, new process)	....do	J. A. Blanchard, New York City.
106	Paris Green	....do	
107	....do	....do	
108	....do	Hatch Experiment Station, Massachusetts, Bulletin No. 81 (1902).	E. & F. King Co., Boston, Mass.
109	Paris Green (composite of 3)	....do	C. T. Reynolds & Co., New York City.
110	Paris Green	....do	I. Pfeiffer, New York City.
111	Paris Green (composite of 2)	....do	J. A. Blanchard, New York City.
112	Paris Green	....do	C. N. Childs & Co., New York City.
113	....do	....do	Leggett & Bro., New York City.
114	....do	....do	A. B. Ansbacher & Co., New York City.
115	....do	....do	Do.
116	Tobacco Liquor	Massachusetts State Experiment Station Report (1890), pp. 275-276.	
117	....do	....do	
118	Paris Green	....do	
119	....do	....do	
120	Sulphatine	....do	
121	Death to Rose Bugs	....do	
122	Prof. De Graff's Bug Destroyer	....do	
123	Paris Green	Massachusetts State Experiment Station Report (1892), p. 336.	
124	....do	....do	
125	....do	....do	

126	.....do	.....do	
127	.....do	.....do	
128	.....do	.....do	
129	Perfect Bug Destroyer	.....do	
130	Tobacco Liquor	.....do	
131	.....do	.....do	
132	Nicotinia	.....do	
133	Hellebore	.....do	
134	.....do	.....do	
135	Peroxide of Silicates	.....do	
136	Oriental Fertilizer and Bug Destroyer	Massachusetts State Experiment Station Report (1893), p. 324.	
137	Nonpoisonous Potato Bug Destroyer	.....do	
138	Paris Green	Mississippi Experiment Station Report (1891), p. 36	Leggett & Co., New York City.
139	.....do	.....do	A. J. Peck, Meridian, Miss.
140	.....do	.....do	Mansfield Drug Co., Memphis, Tenn.
141	.....do	.....do	I. L. Lyons, New Orleans, La.
142	.....do	Montana Experiment Station, Bulletin No. 25 (1900)	John Lucas & Co., New York City.
143	.....do	.....do	
144	.....do	.....do	Acme Color Works, New York City.
145	.....do	.....do	A. B. Ansbacher & Co., New York City.
146	.....do	.....do	F. W. Devoe & Co., New York City.
147	.....do	.....do	C. T. Reynolds & Co., New York City.
148	London Purple	.....do	Do.
149	.....do	.....do	
150	Oriental Fertilizer and Insect Destroyer	New Hampshire Experiment Station Report (1894), pp. 121-122.	
151	Nonpoisonous Insect Exterminator	.....do	
152	Paris Green (average of 5)	New Hampshire Experiment Station, Bulletin No. 36 (1896).	
153	London Purple	.....do	Hemingway's London Purple Co., London, England.
154	Hellebore	.....do	West & Jeremy, Boston, Mass.
155	.....do	.....do	Leggett & Bros., New York City.
156	.....do	.....do	
157	.....do	.....do	
158	.....do	.....do	
159	Paris Green	New Hampshire Experiment Station, Bulletin No. 43	
160	.....do	New Jersey Experiment Station Report (1897), pp. 397-492.	Wetherell & Co.
161	.....do	.....do	John Lucas & Co., New York City.
162	.....do	.....do	
163	.....do	.....do	Cawley, Clark & Co., Newark, N. J.
164	.....do	.....do	C. T. Reynolds & Co., New York City.
165	.....do	.....do	Cawley, Clark & Co., Newark, N. J.
166	.....do	.....do	Robert Shoemaker.
167	.....do	.....do	John Lucas & Co., New York City.
168	Paris Green (Lion brand)	.....do	J. A. Blanchard, New York City.
169	Paris Green (New Process)	.....do	Do.
170	Paris Green	.....do	Leggett & Bros., New York City.
171	Potash Whale-Oil Soap	.....do	Good's.
172	Whale-Oil Soap	.....do	Richardson formula.
173	.....do	.....do	A. Pfeiffer.
174	Whale-Oil Soap (Anchor brand)	.....do	Leggett & Bros., New York City.

TABLE I.—*Source of analysis and description of samples*—Continued.

Sample No.	Name.	Source.	Manufacturer of sample.
175	London Purple	New Jersey Experiment Station Report (1890), p. 523.	
176	X. O. Dust	do	
177	Paris Green (average of 5)	New York Cornell Experiment Station, Bulletin No. 149 (1898).	
178	English Purple	do	
179	Paris Purple	do	
180	do	do	
181	Slug Shot	do	
182	Laurel Green	do	
183	Bug Death	do	
184	Paris Green	New York Cornell Experiment Station, Bulletin No. 177 (1900).	
185	Paragrene	do	
186	Green Arsenite	do	
187	XX	do	
188	Pink Arsenoid	do	
189	Green Arsenoid	do	
190	Green Arsenoid (No. 53)	do	
191	Special Laurel Green	do	
192	Paris Green (average of 24)	New York State Experiment Station, Bulletin No. 165 (1899).	
193	Paragrene	do	
194	do	do	
195	London Purple	do	
196	Laurel Green	do	
197	Electric Vermin Exterminator	do	
198	Paris Green	New York State Experiment Station, Bulletin No. 190 (1900).	Acme Color Works.
199	do	do	Do.
200	do	do	Adler Color and Chemical Works.
201	do	do	Do.
202	do	do	A. B. Ansbacher & Co.
203	do	do	Do.
204	do	do	J. A. Blanchard.
205	do	do	Do.
206	do	do	Cawley, Clark & Co.
207	do	do	C. M. Childs & Co.
208	do	do	Eckstein Bros.
209	do	do	M. Herrmann & Co.
210	do	do	Highlands Chemical Co.
211	do	do	F. L. Lavanburg.
212	do	do	Leggett & Bros.
213	do	do	New York Enamel Paint Co.
214	do	do	John C. Lucas.
215	do	do	I. Pfeiffer.

216	do	do	C. T. Reynolds & Co.
217	do	do	Do.
218	do	do	Do.
219	do	do	
220	Green Arsenoid	do	
221	Paragrene	do	
222	Black Death	do	
223	Bug Death	do	
224	Slug Shot	do	Benj. Hammond, Fishkill on the Hudson, N. Y.
225	Paris Green	New York State Experiment Station, Bulletin No. 204 (1901).	Acme Color Works.
226	do	do	Do.
227	do	do	Adler Color and Chemical Works.
228	do	do	Do.
229	do	do	A. B. Ansbacher & Co.
230	do	do	Do.
231	do	do	Do.
232	do	do	J. A. Blanchard.
233	do	do	Do.
234	do	do	Do.
235	do	do	G. C. Buell & Co.
236	do	do	Cawley, Clark & Co.
237	do	do	C. M. Childs & Co.
238	do	do	Do.
239	do	do	Hampden Paint Co.
240	do	do	Morris Herrmann & Co.
241	do	do	Do.
242	do	do	Do.
243	do	do	Fred L. Lavanburg.
244	do	do	Do.
245	do	do	Do.
246	do	do	Geo. E. Laverack.
247	do	do	Leggett & Bros.
248	do	do	Do.
249	do	do	Do.
250	do	do	
251	do	do	New York Enamel Paint Co.
252	do	do	Do.
253	do	do	Do.
254	do	do	I. Pfeiffer.
255	do	do	Do.
256	do	do	Do.
257	do	do	C. T. Reynolds & Co.
258	do	do	Devoe, Reynolds & Co.
259	do	do	Solomon & Schwartz.
260	do	do	Sondheim, Alsburg & Co.
261	do	do	Stanley, Jordan & Co.
262	do	do	John L. Thompson Sons.
263	do	do	
264	do	do	
265	English Bug Compound	do	English Compound Co.
266	Laurel Green	do	Nichols Chemical Co.
267	do	do	Do.

TABLE I.—*Source of analysis and description of samples*—Continued.

Sample No.	Name.	Source.	Manufacturer of sample.
268	London Purple	New York State Experiment Station Bulletin No. 201 (1901).	Hemingway's London Purple Co.
269	Paragrene	do	F. L. Lavanburg.
270	Paris Green, Bordeaux Mixture	do	Leggett & Bros.
271	Disparene	Ohio Experiment Station, Bulletin No. 127 (1901)	Bowker Chemical Co., Boston, Mass.
272	Arsenate of Lead	do	Swift & Co., Boston, Mass.
273	Paris Green	do	Laubach & Boyd, Wooster, Ohio.
274	do	do	
275	Crude Petroleum	do	
276	do	do	
277	do	do	
278	do	do	
279	Paris Green (average of 18)	Oregon Experiment Station, Bulletin No. 49 (1898)	
280	Fostite	Pennsylvania Experiment Station Report (1892), p. 138.	
281	Par 'oidium	do	
282	Antinonnin	do	
283	Paris Green	South Carolina Experiment Station Report (1888), p. 142.	
284	London Purple	do	
285	Paris Green	Not published; kindly furnished by South Carolina Experiment Station.	
286	do	Vermont Experiment Station Report (1888)	C. T. Reynolds & Co., New York City.
287	London Purple	do	Hemingway's London Purple Co., London, England.
288	Paris Purple	do	A. Poirrier, Paris, France.
289	Peroxid of Silicates	do	Peroxid of Silicate Co., New York City.
290	Slug Shot	do	Hammond's, Fishkill on the Hudson, N. Y.
291	Paris Green	Washington Experiment Station Report (1896) pp. 45–56.	
292	Arsenate of Lead	Not published; data furnished by H. H. Harrington, Texas Agricultural Experiment Station.	F. L. Lavanburg, New York City.
293	do	do	Do.
294	do	do	Merrimac Chemical Co., Boston, Mass.
295	do	do	Do.
296	do	do	Patton-Worsham Drug Co., Dallas, Tex.
297	Paris Green	do	Adler Color and Chemical Co., New York City.
298	do	do	Do.
299	do	do	
300	do	do	
301	do	do	F. L. Lavanburg, New York City.
302	London Purple	do	
303	do	do	
304	Purple Poison	do	Meyer Bros. Drug Co., St. Louis, Mo.
305	Bollene Insecticide	do	Bollene Manufacturing Co., Galveston, Tex.
306	White Arsenic	do	
307	Slug Shot	do	Benj. Hammond, Fishkill on the Hudson, N. Y.
308	Insecticide	do	McLean & Graum, Caldwell, Tex.

23 I & W	Potassium Cyanide	Unpublished data of Bureau of Chemistry U. S. Department of Agriculture.	Elmer & Amend, New York City.
24 I & W	Royal Concentrated Lye	do	Commercial Lye Co., New York City.
26 I & W	Formalin	do	E. Schering, Berlin, Germany.
28 I & W	Copper Carbonate	do	Elmer & Amend, New York City.
63 I & W	Excelsior Insecticide and Wood Preserver (Red Cedar brand).	do	
142 I & W	White Arsenic	do	
143 I & W	do	do	Spotteholz & Ameshot, Amsterdam, Holland.
202 I & W	Fly Preventative	do	
644 I & W	Bollene Insecticide	do	Bollene Manufacturing Co., New York, Baltimore, and Galveston.
645 I & W	do	do	Do.
916 I & W	Karbo-Kresolate	do	J. W. Barwell, Waukegan, Ill.
1170 I & W	Potassium cyanid (99 per cent)	do	Baker, Adamson & Co., Easton, Pa.

PARIS GREENS.

The analyses of a large number of samples of Paris Green are given in Table II, together with notes that aid in explaining the methods of analysis used. Whenever the method of analysis was given in the original publication it is quoted in full, not only in this table, but throughout the bulletin, in order that the reader may judge for himself of its accuracy. In a number of cases in which only one or two constituents were determined in samples of Paris greens the average of all the results is given, mentioning, however, the number that fall below the generally accepted standard for such goods.

METHODS.

The method of determining arsenious oxid in samples Nos. 1 to 6, inclusive, was as follows:

> Weigh 1 gram of the material into a beaker, add 30 cc of strong hydrochloric acid and digest at a temperature below the boiling point, adding small quantities of $KClO_3$ at frequent intervals. Continue heating until the odor of chlorin has disappeared, dilute with water, add ammonia in slight excess, cool, add magnesia mixture drop by drop, stirring vigorously, and let stand twelve hours. Filter, wash with ammonia water, and dry. Detach precipitate from filter and ignite filter, using ammonium nitrate. Transfer the precipitate to a porcelain crucible and heat for a while on an iron plate and finally in the direct flame. Add filter ash and weigh as magnesium pyro-arsenate.

In samples Nos. 37 to 55, inclusive (19 samples), the following method was used for determining soluble arsenious oxid:

> Allow 0.5 gram of the green to stand in suspension in 50 cc of distilled water in a stoppered cylinder for one week, with occasional shaking, and titrate an aliquot filtered portion of this solution for arsenious oxid.

The method for determining total arsenious oxid in samples Nos. 69 to 80, inclusive, was practically the same as that used for samples Nos. 1 to 6, inclusive.

In determining total arsenious oxid in samples Nos. 198 to 264, inclusive (62 samples), the method given in Bulletin 68, Bureau of Chemistry, U. S. Department of Agriculture, for the total arsenious oxid in Paris Green was used. In determining copper the electrolytic method was used. In determining soluble arsenious oxid 1 gram was suspended in 1,000 cc of water and allowed to stand over twenty-four hours in a stoppered flask, with occasional shaking. The arsenious oxid going into solution was titrated with iodin solution.

The soluble arsenious oxid in sample No. 285 was determined by extracting 1 gram of Paris Green with 400 cc of water for nine days, with occasional shaking, and titrating the arsenious oxid in an aliquot portion of the resulting solution with iodin.

TABLE II.—*Composition of Paris greens.*

Sample No.	Moisture.	Total arsenious oxid (As_2O_3).	Total copper oxid (CuO).	Acetic acid.	Sulphur trioxid (SO_3).	Silica (SiO_2).	Sodium oxid (Na_2O).	Calcium oxid (CaO).	Soluble arsenious oxid (As_2O_3).	Arsenious oxid in combination with copper.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
1		55.42									
2		55.01									
3		59.71									
4		53.13									
5		57.38									
6		54.15									
7		0.00	0.00								This sample contained neither copper nor arsenic, but consisted of a mixture of prussian blue, chrome yellow, and either clay or chalk.
8	1.02	54.07	33.46	11.45							
9	.58	55.73	32.54	11.15							
10	2.48	54.38	34.19	8.95							
11	.94	38.21	30.67	30.15							
12		56.18									
13		55.96									
14		54.08									
21	1.31	52.90	31.25	11.28	0.23	0.10	0.77	0.10	23.60		This sample also contained a trace of potassium oxid, 0.58 per cent zinc oxid, and 0.75 per cent of prussian blue.
22		52.94	32.60						29.40		
23		55.13	26.70		Present.		Present.		24.60		The manufacturers of this Paris green claimed 64 per cent arsenious acid and 20 per cent of copper oxid.
24	2.00	48.97	27.93		1.99		1.51		6.70		
25		48.27	28.06		1.30		1.01		8.10		
26		48.68	34.60		.85		.65		9.10		
31	1.34	61.25	33.35	3.93		.13					
34		53.61	29.95								In sample No. 34 the figure for arsenious oxid is an average of 7 Paris greens, one of which contained below 50 per cent arsenious oxid, having only 39.61 per cent. The figure for copper oxid is an average of 2 Paris greens.
37		55.69	31.38						3.82		
38		55.08	31.54						2.44		
39		57.07	31.38						5.52		
40		57.33	31.24						3.93		
41		60.49	30.53						3.92		
42		58.18	31.10						3.89		
43		56.17	29.59						2.86		
44		55.56	30.77						3.52		
45		55.20	29.55						3.58		
46		56.84	31.16						3.30		
47		56.09	30.68						2.80		
48		55.68	31.14						3.11		

TABLE II.—*Composition of Paris greens*—Continued.

Sample No.	Moisture.	Total arsenious oxid (As_2O_3).	Total copper oxid (CuO).	Acetic acid.	Sulphur trioxid (SO_3).	Silica (SiO_2).	Sodium oxid (Na_2O).	Calcium oxid (CaO).	Soluble arsenious oxid (As_2O_3).	Arsenious oxid in combination with copper.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
49		56.84	30.14						3.33		
50		56.96	30.10						2.61		
51		56.72	29.09						2.72		
52		56.78	30.24						2.98		
53		56.48	30.72						3.21		
54		61.05	28.50						4.32		
55		56.23	30.58						2.41		
56		47.53	34.81								
57		29.24	9.78					21.34			
59		49.49	34.18								
60		49.57	32.35								
61		48.64	30.43					.89			
62		56.57	31.02						1.98		
63		56.32	29.23						2.28		
64		58.15	29.76						1.88		
69		52.55									This figure is the average of 10 Paris greens, 2 of which contained less than 50 per cent arsenious oxid, viz, (1) 41.28 per cent, manufactured by C. T. Reynolds & Co., New York City; (2) 49.82 per cent, manufacturer unknown.
70		52.72									
71		50.85									Average of 2 Paris greens, one containing less than 50 per cent arsenious oxid (48.47 per cent, manufactured by Weiland & Meissner).
72		55.91									This figure is the average of 8 Paris greens.
74		54.73									This figure is the average of 39 Paris greens, 1 containing less than 50 per cent arsenious oxid (48.63 per cent, manufacturer unknown).
75		54.17									Average of 39 Paris greens; 1 contained less than 50 per cent arsenious oxid (49.69 per cent, obtained of Martin & Ducote, Lafayette, Iowa).
76		53.97									Average of 36 Paris greens.
77		53.44									Average of 149 Paris greens.
78		54.37									Average of 79 Paris greens.
79		53.17									Average of 35 Paris greens.
80		53.50									Average of 19 Paris greens; 1 contained less than 50 per cent arsenious oxid (49.38 per cent, manufacturer unknown).
81	1.38	54.85	32.55								
82	1.05	54.14	3[illegible]								
83	1.67	53.73	30.60								
84	1.20	56.04	32.30			.40					
85	5.00	58.44	8.30		7.56	1.40		14.50			This sample also contained 0.37 per cent of magnesium oxid.

86	4.29	63.98	6.60		5.80	.60		15.98			This sample also contained 0.56 per cent of magnesium oxid.
87		47.52	8.96		7.47		3.23	15.67			This sample also contained 1.15 per cent of magnesium oxid and 0.04 per cent of potassium oxid.
88	.71	49.70	7.88		5.78		2.36	16.88			This sample also contained 1.37 per cent of magnesium oxid and 0.03 per cent of potassium oxid.
94	.71	57.44	30.59								
95	.75	56.40	30.14								
96	.53	58.57	30.35								
97	.55	56.73	30.62								
98	.56	59.72	30.61								
99	.60	59.38	29.85								
100	.34	58.45	29.26								
101	.60	57.67	30.15								
102	.56	58.08	29.73								
103	.52	57.87	29.49								
104	.55	58.03	30.72								
105	3.72	52.78	21.65								
106	.96	58.06	30.65								
107	.45	58.99	28.44								
108	.60	59.04	30.35			.05					
109	.55	61.15	28.00			.01					This sample is a composite of 3 greens manufactured by the same person.
110	.63	60.38	29.83								
111	.53	60.37	29.51			.14					This sample is a composite of 2 samples manufactured by the same firm.
112	.78	60.71	30.32			.02					
113	.39	60.60	28.95			.15					
114	.86	60.04	29.94			.02					
115		58.52	30.80								
118	1.34	61.25	33.35	3.93		.13					
119	1.31	61.21	33.45	3.94		.09					
123	1.30	62.55	32.84	3.10		.21					
124	1.41	61.40	33.20	3.90		.09					
125	1.40	61.15	33.10	3.71		.64					
126	1.15	59.92	30.40			.10					
127	1.15	53.91	31.27	8.10		.04					
128	1.27	54.80	30.85	6.50		.12					
138	.67	54.48	35.60								
139	.98	54.74	31.23								
140	1.00	56.14	33.30								In reporting samples Nos. 138–141, inclusive, the chemist reported the copper as copper hydrate, added the copper hydrate, moisture, and arsenious acid together and subtracted from 100, reporting the remainder as impurities. The acetic acid was not taken into account.
141	.97	56.46	33.20								
142		57.17	30.26						1.24		
143		59.52	29.98						1.09		
144		60.26	29.68						1.29		
145		57.67	31.08						1.14		
146		57.42	31.36						.84		
147		59.40	30.80						1.09		
152		55.99									This figure is an average of 5 Paris greens.

TABLE II.—*Composition of Paris greens*—Continued.

Sample No.	Moisture.	Total arsenious oxid (As_2O_3).	Total copper oxid (CuO).	Acetic acid.	Sulphur trioxid (SO_3).	Silica (SiO_2).	Sodium oxid (Na_2O).	Calcium oxid (CaO).	Soluble arsenious oxid (As_2O_3).	Arsenious oxid in combination with copper.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
159	...	30.00	...	...	...	43.3	...	...	...	...	The 43.3 per cent, insoluble in hydrochloric acid, consisted of 30.6 per cent silica, 5 per cent oxids of iron, and aluminum and other constituents. This green was evidently composed of about equal parts of Paris Green and earthy matter, such as clay or infusorial earth.
160	...	64.88	...	...	...	...	...	...	...	...	
161	...	47.82	...	...	...	...	...	...	...	...	
162	...	56.19	...	...	...	...	...	...	...	...	
163	...	68.59	...	...	...	...	...	...	...	...	
164	...	58.44	...	...	...	...	...	...	...	...	
165	...	41.54	...	...	...	...	...	...	...	...	
166	...	56.03	...	...	...	...	...	...	...	...	
167	...	63.11	...	...	...	...	...	...	...	...	
168	...	67.38	...	...	...	...	...	...	...	...	
169	...	66.41	...	...	...	...	...	...	...	...	
170	...	59.33	...	...	...	...	...	...	...	...	
177	...	54.73	...	...	...	...	...	...	...	...	This figure is the average of 5 Paris greens.
184	...	56.45	23.62	...	.71	...	...	...	1.83	...	
192	...	56.48	29.97	...	...	...	...	...	...	...	These figures are the averages of 24 Paris greens.
198	...	56.56	30.20	...	...	...	...	...	1.35	...	
199	...	56.50	29.89	...	...	...	...	...	1.10	...	
200	...	56.25	29.85	...	...	...	...	...	1.59	...	
201	...	56.75	30.54	...	...	...	...	...	1.23	...	
202	...	56.81	30.33	...	...	...	...	...	.61	...	
203	...	56.75	29.98	...	...	...	...	...	1.10	...	
204	...	59.20	6.86	...	...	...	...	...	15.69	...	
205	...	58.16	28.53	...	...	...	...	...	.74	...	
206	...	56.62	31 20	...	...	...	...	...	.74	...	
207	...	57.17	30.33	...	...	...	...	...	.74	...	
208	...	57.11	28.76	...	...	...	...	...	1.10	...	
209	...	56.62	30.16	...	...	...	...	...	1.23	...	
210	...	57.11	30.39	...	...	...	...	...	.98	...	
211	...	56.50	30.16	...	...	...	...	...	.86	...	
212	...	56.93	30.39	...	...	...	...	...	1.10	...	
213	...	56.75	29.45	...	...	...	...	...	1.59	...	
214	...	55.83	30.73	...	...	...	...	...	.98	...	
215	...	56.75	30.16	...	...	...	...	...	.74	...	
216	...	60.80	27.22	...	...	...	...	...	.98	...	
217	...	57.05	30.70	...	...	...	...	...	.86	...	
218	...	56.75	30.39	...	...	...	...	...	.86	...	
219	...	56.17	30.23	...	...	...	...	...	.86	...	

225	....	57.11	30.04	....	....	....	....	....	1.47	56.27	The amount of arsenious oxid in combination with copper is calculated by assuming that all copper present is combined except when formed in excess. This calculation is necessary because the present New York law requires that Paris Green shall contain arsenic in combination with copper equivalent to not less than 50 per cent of arsenious oxid.
226	....	57.66	30.04	....	....	....	....	....	1.47	56.27	
227	....	57.97	30.29	....	....	....	....	....	.88	56.74	
228	....	56.93	30.10	....	....	....	....	....	2.64	56.88	
229	....	57.11	30.23	....	....	....	....	....	.88	56.63	
230	....	57.84	30.48	....	....	....	....	....	1.47	57.10	
231	....	56.93	30.35	....	....	....	....	....	1.10	56.85	
232	....	57.42	28.54	....	....	....	....	....	1.53	53.46	
233	....	57.11	29.22	....	....	....	....	....	1.53	54.73	
234	....	57.35	29.66	....	....	....	....	....	1.53	55.56	
235	....	57.72	29.41	....	....	....	....	....	1.65	55.09	
236	....	57.42	31.23	....	....	....	....	....	1.35	57.42	
237	....	57.85	30.08	....	....	....	....	....	1.23	56.85	
238	....	58.76	30.41	....	....	....	....	....	.98	56.97	
239	....	58.82	30.16	....	....	....	....	....	1.10	56.50	
240	....	61.40	27.47	....	....	....	....	....	1.78	51.46	
241	....	62.87	26.78	....	....	....	....	....	2.21	50.17	
242	....	62.69	26.53	....	....	....	....	....	1.53	49.70	
243	....	57.91	30.16	....	....	....	....	....	.98	56.50	
244	....	57.91	30.48	....	....	....	....	....	.98	57.10	
245	....	58.82	29.91	....	....	....	....	....	1.15	56.03	
246	....	58.03	29.85	....	....	....	....	....	1.23	55.91	
247	....	57.11	30.01	....	....	....	....	....	.98	56.21	
248	....	56.13	29.54	....	....	....	....	....	1.23	55.34	
249	....	57.42	30.50	....	....	....	....	....	1.23	57.13	
250	....	57.42	30.41	....	....	....	....	....	.98	56.97	
251	....	57.11	30.04	....	....	....	....	....	1.47	56.27	
252	....	58.09	30.23	....	....	....	....	....	1.72	56.63	
253	....	58.46	30.35	....	....	....	....	....	.98	56.85	
254	....	57.35	30.04	....	....	....	....	....	1.10	56.27	
255	....	57.72	30.73	....	....	....	....	....	.88	57.57	
256	....	58.09	30.29	....	....	....	....	....	1.35	56.74	
257	....	60.72	28.35	....	....	....	....	....	1.40	53.11	
258	....	57.29	30.54	....	....	....	....	....	1.10	57.21	
259	....	57.60	30.85	....	....	....	....	....	.88	57.60	
260	....	57.84	30.48	....	....	....	....	....	.88	57.10	
261	....	58.58	29.60	....	....	....	....	....	1.40	55.45	
262	....	57.60	30.29	....	....	....	....	....	.88	56.71	
263	....	57.72	31.14	....	....	....	....	....	.98	57.72	
264	....	58.09	30.16	....	....	....	....	....	.98	56.50	
273	....	58.53	30.92	8.00	....	....	....	....	....	....	
274	....	58.99	28.95	7.50	....	....	....	....	....	....	
279	....	54.43	29.47	....	....	....	....	....	....	....	The figure 54.53 is the average of 18 Paris greens, 8 of which are below 50 per cent arsenious oxid (viz. 46.86 per cent, 37.80 per cent, 45.51 per cent, 45.08 per cent, 40.98 per cent, 46.16 per cent, 34.10 per cent, 46.60 per cent; manufacturers unknown). The figure 29.47 is the average of 12 Paris greens.
283	1.88	40.36	....	....	....	....	....	....	....	....	
285	....	56.00	30.32	....	....	....	....	....	2.88	....	
286	1.10	57.08	30.40	11.42	....	Trace.	....	....	....	....	
291	....	57.06	....	....	....	....	....	....	....	....	

TABLE II.—*Composition of Paris greens*—Continued.

Sample No.	Moisture.	Total arsenious oxid (As_2O_3).	Total copper oxid (CuO).	Acetic acid.	Sulphur trioxid (SO_3).	Silica (SiO_2).	Sodium oxid (Na_2O).	Calcium oxid (CaO).	Soluble arsenious oxid (As_2O_3).	Arsenious oxid in combination with copper.	Remarks.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	
297	....	55.90	29.77	....	....	....	....	....	0.95	....	Analysis made in accordance with Texas law regulating sale of Paris greens and other insecticides and is only good until May 1, 1903.
298	....	57.25	30.00	....	....	....	....	....	.92	....	Analysis made under Texas law; good only until May 1, 1902.
299	....	55.15	29.74	....	....	....	....	....	.76	....	Do.
300	....	57.63	31.20	....	....	....	....	....	.60	....	Do.
301	....	55.45	29.65	....	....	....	....	....	1.00	....	Do.

COMMENT ON ANALYSES.

A glance at Table II at once makes it apparent that only 1 sample out of more than 650 examined contains neither copper, acetic acid, nor arsenious oxid—this is sample No. 7. One other sample, No. 159, consists of about equal parts Paris Green and some insoluble material that appears to be clay or infusorial earth. Out of the total number examined 31 contained less than 50 per cent arsenious oxid. It will be noted, however, that a number of these samples said to contain less than 50 per cent arsenious oxid were analyzed by a method (the magnesium pyro-arsenate method) that has since been shown to give results about 1 per cent low. If a more accurate method of analysis had been used, the number falling below 50 per cent arsenious oxid would very likely be materially diminished. It also appears that the Western States receive more of those samples that contain small percentages of arsenious oxid than do the Northern States.

The State of Louisiana, which previous to 1899 occasionally received samples containing small percentages of arsenious oxid, does not seem to have had any upon the market during 1899 and 1900. Out of 96 samples of Paris Green examined in New York State during the years 1899 to 1901, inclusive, none was found that contained less than 50 per cent arsenious oxid. In both California and Oregon, until a very recent date, quite a large amount of the Paris Green on the market contained small percentages of arsenious oxid, although it is said that this condition has been very much improved during the last two years.

Concerning soluble arsenious oxid in Paris greens, it appears that those upon the market in California until recently contained very large quantities, those in Idaho were very fair, only 2 out of 19 containing more than 4 per cent, those in Illinois, Montana, and Texas contained very small percentages, and those in New York the same, only 1 out of 90 containing more than 4 per cent. This one was sample No. 204, containing 15.69 per cent of free arsenious oxid.

It must be borne in mind, however, in looking over the figures for free arsenious oxid that the results in one State can not be compared with those in another, since the methods of analysis were very likely different in every case and would lead to entirely different results. It may be of interest to those who have not read the previous bulletin of the Bureau of Chemistry on this subject to mention that out of the 45 samples of Paris Green, from all parts of the United States, which were examined 71 per cent contained more than 4 per cent of soluble arsenious oxid and 16 per cent contained more than 6 per cent of arsenious oxid when a ten-day extraction of 1 gram with 1,000 cc of water was made. In view of the facts brought out in that bulletin, a 6 per cent, rather than a 4 per cent, limit for soluble arsenious oxid was advocated when the above method of analysis was employed.

From the figures given above, together with information received from the various States, it appears that upon the whole the purity of the Paris greens upon the American market is steadily and rapidly upon the increase.

LONDON, ENGLISH, AND PARIS PURPLES.

The analyses of 10 samples of London Purple, 1 sample of English Purple, and 3 samples of Paris Purple are given in the following table (III). All of these are products obtained by boiling a waste product from the analine-dye industry with lime. Since it was not known at the time these analyses were made that the arsenic was present, both as arsenious and arsenic acids, the total arsenic was determined and reported as arsenious oxid, only the soluble arsenious oxid being determined, instead of both soluble arsenic and arsenious oxids.

TABLE III.—*Composition of London purples and closely allied mixtures.*

Sample No.	Name.	Moisture.	Arsenious oxid (As_2O_3).	Calcium oxid (CaO).	Ferric oxid and alumina (Fe_2O_3 and Al_2O_3).	Sulphur trioxid (SO_3).	Dye.	Soluble arsenious oxid (As_2O_3).
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
148	London Purple		51.43					0.54
149	do		37.18					2.58
153	do		37.56					
175	do	3.27	41.44	24.32	3.87	0.81	27.29	(a)
195	do		32.88					
268	do		32.32					12.21
284	do	3.99	42.60					
287	do	3.37	46.73	24.35	1.31	[b] .46		
302	do		31.90	[c] 25.78				
303	do		31.50	[d] 25.00				
178	English Purple		36.75					5.36
179	Paris Purple		47.05					11.86
180	do		34.10					13.88
288	do	6.05	33.72	4.55	4.76	[e] .91		

[a] A portion digested 40 hours with water at room temperature gave 8.11 per cent arsenious oxid and 2.85 per cent soluble calcium oxid. A portion digested with hot water until all the soluble arsenious oxid had dissolved gave 16.45 per cent soluble arsenious oxid and 3.26 per cent soluble calcium oxid.

[b] Also contains 1.65 per cent nitrogen.

[c] This analysis was made in accordance with the Texas law regulating the sale of Paris Green and other insecticides and is good only until May 1, 1903.

[d] Made in accordance with Texas law, but good only until May 1, 1902.

[e] Also contained 2.8 per cent nitrogen and 0.57 per cent potassium oxid.

It will at once be noted that the variation in the amount of arsenic in different samples of London Purple and Paris Purple is very large. In the case of 11 samples examined more recently in this Bureau (published in Bulletin 68) this variation in the arsenic content is not nearly so great. It would, therefore, appear that the amount of this constituent in such goods is becoming more constant in the more recently manufactured samples.

The figures for soluble arsenious oxid can not be compared with those obtained more recently in this laboratory, since the soluble arsenic oxid was not determined in the earlier analyses, and it is only when both figures are at hand that any conclusions of a positive nature can be drawn.

INSECTICIDES AND FUNGICIDES, OTHER THAN PARIS GREEN AND LONDON PURPLE, THAT CONTAIN ARSENIC, COPPER, OR BOTH, AS THEIR ACTIVE CONSTITUENTS.

In Table IV is given a compilation of analyses of Green Arsenoid, Green Arsenite, Paragrene, Laurel Green, and White Arsenic, together with two analyses of White Arsenic made in this laboratory.

TABLE IV.—*Composition of Green Arsenoid, Green Arsenite, Paragrene, Laurel Green, and White Arsenic.*

Sample No.	Name.	Moisture.	Total arsenious oxid (As_2O_3).	Copper oxid (CuO).	Calcium oxid (CaO).	Sulphur trioxid (SO_3).	Soluble arsenious oxid (As_2O_3).
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
29[a]	Green Arsenoid	2.77	61.43	28.83			7.82
90	do	1.44	50.77	31.90			
189	do		60.63	29.29		1.55	3.53
190	do. [No. 53]		53.71	29.43		3.70	8.33
220	do	1.91	58.82	30.76			2.94
186	Green Arsenite		54.10	31.59		3.70	2.36
30[b]	Paragrene	6.20	40.60	23.46			23.08
58	do		19.31	19.92	13.81		
185	do		55.57	27.68		1.34	3.21
193	do		43.84	18.08			
194	do		52.30	21.64			
221	do	8.15	36.11	17.87	14.20		1.47
92	Laurel Green	7.64	7.34	13.50	26.31		
182	do		7.75	12.52			
191	Laurel Green (special)		42.69	4.50	35.39		1.15
196[c]	Laurel Green		3.83	11.50			
266	do		4.85	12.68			.00
267	do		5.45	12.05			.00
306[d]	White Arsenic		98.00				
142 (I. & W.)[e]	do		99.86				
143 (I. & W.)[e]	do		99.99				

[a] The following constituents were also present: Silica, 0.40 per cent; organic matter, 6.67 per cent.

[b] In sample No. 30 the following constituents were also present: Acetic acid, 6.72 per cent; calcium sulphate, 19.31 per cent; sodium sulphate, 2.26 per cent; sodium chlorid, 0.25 per cent; ferric oxid, 0.20 per cent.

[c] Large amounts of calcium carbonate and hydrate were also present.

[d] This analysis was made in accordance with the Texas law regulating the sale of Paris Green and other insecticides and is good only until May 1, 1902.

[e] The method of analysis used is that described for total arsenious oxid in Paris Green in Bulletin 68 of this Bureau.

The figures obtained by different analysts for Green Arsenoid, Paragrene, and Laurel Green show that the composition of these substances is very variable, especially as to the content of arsenious oxid, which varies in the first case from 50.77 per cent to 61.43 per cent, in the second from 19.31 per cent to 55.57 per cent, and in the third from 3.83 per cent to 42.69 per cent. The figures obtained for soluble arsenious oxid, here as elsewhere in this compilation, can not be compared, since the methods of extraction were very likely different.

The two samples of White Arsenic analyzed in this laboratory were received from Boise City, Idaho, by the Division of Entomology. They are both evidently of exceptional purity and will give good results when used for preparing Calcium Arsenite, the purpose for which they were purchased.

WHITE ARSENOIDS.

TABLE V.—*Composition of White Arsenoids.*

Constituents.	Sample No. 27.	Sample No. 91.
	Per cent.	*Per cent.*
Barium oxid	42.39	41.43
Barium to join with chlorin	8.60	6.16
Lead oxid	1.55	.96
Carbon dioxid	10.15	8.92
Chlorin	4.45	3.19
Silica	.20	....
Arsenious oxid	27.64	31.90
Moisture	4.00	2.35
Soluble arsenious oxid	27.64	....

PARIS GREEN AND BORDEAUX MIXTURE.

The composition of this sample, No. 270, was as follows:

	Per cent.
Total arsenious oxid	15.49
Copper oxid	16.02
Soluble arsenious oxid	1.72

SLUG SHOTS.

TABLE VI.—*Composition of Slug Shots.*

Constituents.	Sample No. 181.	Sample No. 290.	Sample No. 307.
	Per cent.	*Per cent.*	*Per cent.*
Arsenious oxid	0.60	1.58	....
Copper oxid	1.44	.60	....
Lime	....	30.25	28.82
Sulphur trioxid	....	43.02	41.18
Ferric oxid and alumina	....	.79	....
Sodium chlorid	....	2.83	....
Insoluble	....	1.38	....
Volatile organic matter (by difference)	....	4.79	....

The sodium chlorid in sample No. 290 was calculated from a content of 1.72 per cent of chlorin. The volatile organic matter in this sample consisted principally of dead oil.

In sample No. 307 small amounts of sulphur, copper sulphate, oxid of iron, carbolic acid, and tobacco, with less than 1 per cent of arsenic, were also present.

Slug Shot is evidently a mixture of fairly constant composition containing in all cases small amounts of arsenious oxid and copper oxid as active poisonous constituents.

PEROXID OF SILICATES.

TABLE VII.—*Composition of peroxid of silicates.*

Constituents.	Sample No. 135.	Sample No. 289.
	Per cent.	*Per cent.*
Moisture	1.65	1.44
Arsenious oxid	.57	1.47
Copper oxid	.33	.28
Sulphur trioxid	49.66	52.29
Calcium oxid	41.18	36.43
Insoluble	2.31	1.98
Ferric oxid and alumina	....	1.40
Organic and volatile matter (by difference)	....	4.76

This mixture evidently contains arsenious oxid and copper as the active constituents, diluted with a large amount of gypsum. In so far as the arsenious oxid and copper content are concerned, this mixture is of value as an insecticide; but the gypsum is inert in its action upon insects, except that when the spiracles are not well protected they may be closed up with this or any other fine dust.

BLACK DEATH.

Composition of Black Death.

[No. 222.]

	Per cent.
Moisture	9.78
Sulphate of lime (gypsum)	45.34
Magnesium oxid	3.98
Ferric oxid and alumina	3.02
Arsenious oxid	.79
Copper oxid	.41
Silica	5.42
Loss on ignition	28.91

ENGLISH BUG COMPOUND.

The composition of this compound, No. 265, is as follows:

	Per cent.
Arsenious oxid	1.46
Copper oxid	.60

ORIENTAL FERTILIZER AND BUG DESTROYER.

[No. 136.]

Composition of Oriental Fertilizer and Bug Destroyer.

	Per cent.
Water	37.14
Total solids	12.86
Arsenic oxid	2.38
Potassium oxid	3.50
Sodium oxid	6.08
Nitrogen	.67
Chlorin	3.00
Sulphur trioxid	.64

This mixture is evidently sodium and potassium arsenates dissolved in water. In so far as its content of arsenic is concerned, it would be of value in killing insects. It might, however, if sprayed upon trees, cause some damage by scorching, as the arsenic is in a soluble condition. Its fertilizing value is shown by its content of potash and nitrogen.

Another sample of Oriental Fertilizer and Bug Destroyer, No. 150, gave, in 1,000 cc of liquid, 13.12 grams of arsenic and 10.28 grams of potassium oxid.

LEAD ARSENATE AND PINK ARSENOID.

The analyses of samples of Lead Arsenate and Pink Arsenoid, or Lead Arsenite, are given in the following table:

TABLE VIII.—*Composition of Lead Arsenate and Pink Arsenoid.*

Sample No.	Name.	Moisture.	Lead oxid (PbO).	Arsenic oxid (As_2O_5).	Arsenious oxid (As_2O_3).	Soluble arsenious oxid (As_2O_3).
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
[a] 271	Disparene	40.40	38.50	13.28	....	....
[b] 272	Lead Arsenate	36.61	39.14	14.88	....	....
[c] 292	.....do	29.00	40.60	....	[d] 21.20	....
[c] 293	.....do	[f] 35.30	32.34	....	[d] 30.00	....
[e] 294	.....do	[f] 31.50	44.40	....	[d] 18.00	....
[e] 295	.....do	[f] 37.40	44.98	....	[d] 14.70	....
[e] 296	.....do	32.92	34.83	....	[d] 30.62	....
[g] 28	Pink Arsenoid	.31	49.58	....	43.26	3.24
89	.....do	.35	53.83	....	40.16	....
188	.....do	....	49.09	....	49.19	1.93

[a] All the lead oxid calculated to $Pb_3(AsO_4)_2$ leaves an excess of 0.05 per cent arsenic oxid, probably combined as sodium arsenate.

[b] All the lead oxid calculated to $Pb_3(AsO_4)_2$ leaves an excess of 1.38 per cent arsenic oxid, probably combined as sodium arsenate.

[c] This analysis was made in accordance with the Texas law regulating the sale of Paris Green and other insecticides and is good only until May 1, 1903. In addition to constituents named, 2.7 per cen organic matter was found.

[d] The chemist reported arsenic in the following ways on these 5 samples: (1) White arsenic, (2) total arsenic, (3) white arsenic, (4) white arsenic, (5) arsenious oxid. These terms are usually applied to arsenic when in the "ous" condition and mean so much As_2O_3. If these compounds are arsenates, as claimed, the arsenic should be reported as As_2O_5. In several of the above analyses, however, if the figures as given were calculated to As_2O_5, the sum total of the constituents would be markedly above 100 per cent.

[e] Analysis made according to Texas law, but good only until May 1, 1902.

[f] Includes organic matter also.

[g] In addition to constituents named 6.85 per cent of organic matter, lead sulphate, etc., was found

It is at once evident that the variation in the composition of samples of Lead Arsenate is considerable, depending to a large extent on the variation in the moisture content and somewhat on the variation in method of manufacture. These differences in composition should be borne in mind in using such compounds for spraying purposes, since some of them would not have the same insect-killing powers as others at the same dilution. Pink Arsenoid, although somewhat variable in composition, is much more constant than is Lead Arsenate.

XX, PURPLE POISON, AND INSECTICIDE.

TABLE IX.—*Composition of XX, Purple Poison, and Insecticide.*

Constituents.	XX (No. 187).	Purple Poison (No. 304).[a]	Insecticide (No. 308).[a]
	Per cent	*Per cent.*	*Per cent.*
Total arsenious oxid	33.71	30.6	8.00
Calcium oxid	....	8.5	....
Lead oxid (combined)	....	....	15.90
Organic matter (fixed)	....	....	66.60
Organic matter (volatile)	....	....	9.50
Soluble arsenious oxid	32.23	9.5	8.00

[a] The Purple Poison, sample No. 304, also contains coloring matter. The two analyses, Nos. 304 and 308, were made in accordance with the Texas law regulating the sale of Paris Green (see page 61) and other insecticides and holds good only until May 1, 1903.

None of the three analyses is sufficiently complete to show definitively the chemical composition of the compound examined, although from the data as given the Purple Poison appears to be at least partially composed of London Purple or some closely allied product.

COPPER CARBONATE.

[No. 28 I and W.]

The analysis of this sample in the Bureau of Chemistry showed that it contained 58.24 per cent of total copper oxid. It is basic in character and of somewhat variable composition, so that the purity can not be calculated from the copper-oxid content. It contains, however, a somewhat smaller amount of copper oxid than other samples examined in this laboratory. The method of analysis used was that described in Bulletin 68.

FOSTITE.

Composition of fostite.

[No. 280.]

	Per cent.
Loss at dull-red heat	2.92
Soluble in aqua regia:	
Copper oxid	2.98
Ferric oxid and alumina	1.36
Calcium oxid	2.31
Magnesium oxid	1.51
Sulphur trioxid	3.28
Silica	.35
Insoluble in aqua regia	83.77
Soluble in hot water	7.49

This material is said by the analyst to be composed of copper sulphate and finely powdered soapstone in the ratio of approximately 1:9.

SULPHATINE AND DEATH TO ROSE BUGS.

TABLE X.—*Composition of Sulphatine and Death to Rose Bugs.*

Constituents.	Sulphatine (No. 120).	Death to Rose Bugs (No. 121).
	Per cent.	*Per cent.*
Moisture	1.40	2.95
Calcium oxid	18.60	17.76
Copper oxid	2.61	1.05
Sulphur trioxid	4.73	4.35
Sulphur	48.28	34.53
Insoluble	1.63	.49
Ash	....	54.14

Neither of the above substances has ever been examined in this laboratory, but from the analyses it would appear that sulphur and copper, probably as copper sulphate, are the active constituents. These are diluted with inert materials, probably to mitigate their action upon the foliage.

SOAPS.

TABLE XI.—*Composition of whale-oil soaps.*

Sample No.	Moisture.	Potash lye.	Soda lye.
	Per cent.	*Per cent.*	*Per cent.*
171	27.30	10.55	7.54
172	71.58	6.86	4.89
173	6.22	13.86	9.90
174	6.25	10.66	7.61

The above analyses do not indicate that both soda and potash are present, but show how much of either would be present calculated from the number of cubic centimeters of normal acid necessary to neutralize a known weight of soap.

LYES.

The compiled analyses of a number of samples of lyes and the results on one sample (No. 24 I & W) examined in this laboratory are given in Table XII.

TABLE XII.—*Composition of lyes.*

Sample number.	Name.	Moisture.	Sodium hydrate (NaOH).	Potassium carbonate (K_2CO_3).	Sodium chlorid (NaCl).	Sodium carbonate (Na_2CO_3).	Sodium sulphate (Na_2SO_4).	Potassium hydrate. (KOH).	Potassium sulphate. (K_2SO_4)
		Per ct.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per ct.*	*Per ct.*
15	Solid Lye	6.4	22.7	....	66.6	....	....	....	....
16	Concentrated Lye	.5	80.4	....	5.9	....	13.2	....	....
17	Double Concentrated Caustic Soda	......	99.3	....	....	....	....	....	....
18	Refined Pearl Ashes	23.8	....	69.1	....	....	....	....	....
19[a]	Commercial Potash	1.5	....	16.8	4.9	....	14.8	52.3	7.5
20[b]	Canadian Crude Potash	4.5	....	71.4	....	2.3	....	....	14.4
32	Concentrated Potash Lye	.58	86.90	....	7.56	....	4.85	....	....
33[c]	Concentrated Lye	1.87	72.01	....	16.70	.33	4.94	....	....
24 (I & W).	Royal Concentrated Lye	......	70.96	....	....	3.17	....	....	....

[a] The following constituents were also determined: 2.1 per cent insoluble, and 0.1 per cent silica.

[b] The following constituents were also determined: 3.6 per cent potassium chlorid, and 2.7 per cent insoluble.

[c] The following constituents were also found: 0.06 per cent of alumina and traces of ferric oxid, magnesium oxid, and insoluble.

The method used for the analysis of the Royal Concentrated Lye (No. 24 I & W) is as follows:[a]

The lye is weighed in a weighing bottle, dissolved to a definite volume in carbon-dioxid-free water and aliquot portions taken for analysis. Phenolphthalein is added to one of these portions and N/3 potassium acid sulphate is added at the rate of about a drop per second, with constant stirring until the pink color fades and the solution becomes colorless. The number of cc of N/3 acid used, "n" represents the sodium hydroxid and one-half the sodium carbonate, since the sodium carbonate is changed to sodium bicarbonate ($NaHCO_3$). A drop of methyl orange is added and the titration continued until the appearance of a pink color. Let this number of cc be represented by "m." Since this last titration represents the sodium bicarbonate present or one-half the sodium carbonate, "2 m" represents all the sodium carbonate, and "n–m" the sodium hydroxid.

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[a] Cameron. U. S. Dept. of Agr., Div. of Soils Bul. 18.

The figures in most cases explain themselves, although a few remarks emphasizing certain points might not be out of place. Sample No. 15 is evidently a very poor example of a soda lye, as two-thirds of it is composed of sodium chlorid. No. 16 is a very fair sample of caustic soda, while No. 17 is a most excellent sample of the same goods. No. 32 is not a potash lye, as it claims to be, but a soda lye. Nos. 32, 33, and 24 I & W are all fair samples of commercial soda lye.

HELLEBORES.

TABLE XIII.—*Composition of hellebores.*

Sample No.	Ash insoluble in hydrochloric acid.	Total ash.	Sample No.	Ash insoluble in hydrochloric acid.	Total ash.
	Per cent.	*Per cent.*		*Per cent.*	*Per cent.*
133	2.34		156		12.00
134	38.12		157		10.60
154		33.13	158		34.00
155		16.00			

Samples Nos. 134, 154, and 158 are evidently badly contaminated with dirt, probably because the roots of the plant were not properly cleaned. Sample No. 155 also appears to have a slightly higher ash content than should be allowed in preparations of this kind.

MIXTURES CONTAINING BORAX.

A single example of this class of compounds, the analysis of which was made in this laboratory, is given below:

HOOKER'S COCKROACH AND WATER BUG EXTERMINATOR.

Composition of Hooker's Cockroach and Water Bug Exterminator.

	Per cent.
Camphor	3.5–4.0
Borax ($Na_2B_4O_7 10\ H_2O$)	92.25
Ammonia (NH_3)	.27

The remainder is principally organic matter and appears to be tobacco. The ammonia seems to be present as ammonium carbonate.

In making an analysis of this compound the camphor was determined by extracting the powder with ether, allowing it to evaporate at room temperature and weighing at once. While this method leaves much to be desired, it at least gives approximate results when a large portion of the material is used. The borax was determined by the same method as given in Bulletin 68 for similar compounds, except that the ammonia was all driven off from the solution by boiling before the boric acid was determined.

This mixture would very likely be efficacious in driving away cockroaches and water bugs, but probably it would not kill them.

TOBACCO EXTRACTS.

The analyses of five samples of tobacco extracts are given in Table XIV. In two of these only the most important constituent, nicotin, was determined, while in one of them the determination of this constituent was not made.

TABLE XIV.—*Composition of tobacco extracts.*

Constituents.	Sample No. 116.[a]	Sample No. 117.[b]	Sample No. 130.	Sample No. 131.	Sample No. 132.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Moisture	37.710	40.890	....	....	10.00
Ash	19.420	27.770	....	....	....
Total nitrogen	2.010	1.730	....	....	....
Nitrogen as nitrates	.170	....	....	....	....
Nicotin	2.115	.530	4.55	4.82	....
Ferric oxid and alumina	.229	.017	....	....	....
Calcium oxid	3.069	1.466	....	....	4.45
Magnesium oxid	2.303	1.121	....	....	....
Phosphorus pentoxid	.404	.057	....	....	....
Sodium oxid	.207	.525	....	....	....
Potassium oxid	6.550	16.340	....	....	9.15
Insoluble	Trace.	....	....	....	2.12

[a] Specific gravity, 1.3858.

[b] Specific gravity, 1.3777.

In the first four of these samples the nicotin figure is much too low for a good tobacco extract; especially is this true of the first two.

FORMALINS.

The analysis of three samples of formalin, and the average of the results obtained on five samples, together with one analysis made in this laboratory, are given in the following table:

TABLE XV.—*Composition of formalins.*

Sample No.	Formaldehyde.	
	By weight.	By volume.
	Per cent.	*Per cent.*
65	36.9	40.8
66	37.0	40.0
67	38.9	41.9
68	[a] 36.69	....
26 (I & W)	37.18	....

[a] Average of 5 samples.

The method used for determining formaldehyde in samples 65 to 68, inclusive, is as follows:[a]

Fifteen cubic centimeters of N/10 $AgNO_3$ are treated with 5 drops of 50 per cent nitric acid in a 50 cc flask. Ten cc of a solution of potassium cyanid (containing 3.1 grams of 96 per cent potassium cyanid in 500 cc of water) are then added, the flask filled to the mark with distilled water, well shaken, and an aliquot portion of the filtrate, say 25 cc, titrated according to the method of Volhart with a N/10 solution of ammonium sulphocyanate for the excess of silver.

Another 15 cc portion of N/10 $AgNO_3$ is treated with 5 drops of 50 per cent nitric acid and 10 cc of the potassium cyanid solution, to which has been added a known amount of the formalin solution. The whole is made up to 50 cc, and a 25 cc filtrate from it titrated with N/10 ammonium sulphocyanate, the same as before for the excess of silver. The difference between these two results gives the amount of potassium cyanid that has been used by the formaldehyde in terms of N/10 ammonium sulphocyanate, and since each cubic centimeter of this is equal to 3 milligrams of formaldehyde, we can obtain the amount of formaldehyde that has been acted upon by the potassium cyanid.

The method used in the analysis of sample No. 26, examined in this laboratory, is as follows:

About 3 grams of the solution is weighed into a tall Erlenmeyer flask containing 25 to 30 cc of double normal sodium hydroxid. Fifty cc of pure 2.5 to 3 per cent fresh hydrogen peroxid is then gradually added, during a space of three minutes, through a funnel placed in the neck of the flask to prevent spurting. After standing ten minutes the funnel is washed with water and the unused sodium hydroxid titrated with double normal sulphuric acid, using litmus as indicator. By this method the formaldehyde is oxydized to formic acid, which is neutralized by the sodium hydroxid present.

While samples Nos. 65, 66, 67, and 26 I & W all contain less than 40 per cent formaldehyde by weight they contain 40 per cent or over by volume, so that the manufacturers are perfectly correct in claiming that their goods contain as much as 40 per cent of formaldehyde.

PETROLEUMS.

The results obtained by fractionally distilling four samples of crude petroleum are given in the following table:

TABLE XVI.—*Composition of crude petroleum.*

Temperature.	Character of distillate obtained.	Sample No. 275.[b]	Sample No. 276.[c]	Sample No. 277.[d]	Sample No. 278.[c]
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Below 50° C					0.85
50°-70° C	Petroleum ether				2.06
70°-90° C	Gasoline				1.00
Below 80° C	Light naphtha	1.49	0.14	1.89	
80°-120° C	Heavy naphtha	4.35	1.63	9.26	
90°-120° C	Naphtha				5.12
120°-150° C	Benzine	5.03	3.82	9.04	5.35
150°-200° C	Light burning oils	7.64	13.48	10.73	13.70
200°-250° C	Heavy burning oils	13.54	12.03	15.80	12.49
250°-300° C	Illuminating oils	68.70	68.62	45.60	17.76
300°-315° C	do				23.98
315° C+residue				7.70	16.98

[a] Zeit für anal. Chemie, 1897, p. 19.
[b] Specific gravity, 35° Baumé.
[c] Specific gravity, 34° Baumé.
[d] Specific gravity, 45° Baumé.

MISCELLANEOUS SOLID INSECTICIDES AND FUNGICIDES.

The analyses of a number of solid insecticides and fungicides that do not come under any of the previous headings are given in the following tables:

SULPHURS.

TABLE XVII.—*Composition of sulphurs.*

Constituents.	Sample No. 35.	Sample No. 36.
	Per cent.	*Per cent.*
Soluble sulphur	99.960	98.660
Insoluble sulphur	.144	.576
Lime	.000	Trace.
Foreign matter	.196	.764
Total	100.300	100.000

The total for sample No. 35 is given by the chemist who made the analysis as 100 per cent, but all the constituents added together amount to 100.3 per cent, as above.

PINE TAR INSECTICIDES.

[No. 73.]

This is an insecticide obtained from the destructive distillation of pine knots. It readily mixes with water and is slightly sticky and greasy.

BUG DEATHS.

TABLE XVIII.—*Composition of bug deaths.*

Constituents.	Sample No. 93.	Sample No. 223.
	Per cent.	*Per cent.*
Moisture	0.03	0.25
Lead oxid	1.58	2.01
Zinc oxid	78.86	86.80
Ferric oxid and alumina	3.80	5.20
Silica		2.96
Loss on ignition		2.43
Phosphoric acid		.03
Nitrogen		.04
Potash		.00

POTASSIUM CYANID.

[Nos. 23 and 1170 I & W.]

Sample No. 23 contained 53.12 per cent of potassium cyanid calculated from the cyanogen. The remainder of the sample appeared to consist principally of moisture and some potassium carbonate and chlorid. The method of analysis used was that described in Bulletin 68, Bureau of Chemistry.

Composition of potassium cyanid.

[No. 1170, I & W.]

	Per cent.
Cyanogen	40.49
Potassium	21.48
Sodium	23.74
Chlorin	8.98
Carbonic acid radical	1.98
Moisture	.82
Undetermined	2.51
	100.00

The above analyses show that both samples of potassium cyanid are very impure, although the second sample contains just about the amount of cyanogen demanded by theory.

NONPOISONOUS POTATO-BUG DESTROYER.

Composition of nonpoisonous potato-bug destroyer.

[No. 137.]

	Per cent.
Moisture	0.00
Nitrogen	.08
Ash	79.85
Potassium oxid	.00
Calcium oxid	68.20
Magnesium oxid	7.29
Ferric oxid and alumina	1.38
Phosphorus pentoxid	Trace.
Insoluble before calcination	7.29
Insoluble after calcination	1.50

As this analysis is not complete, no opinion as to the substances of which the mixture is composed can be expressed.

NONPOISONOUS INSECT EXTERMINATOR.

[No. 151.]

This sample contained 78.12 per cent of lime and 3.61 per cent crude carbolic acid, determined as ether extract. It had an odor of carbolic acid and appeared to be partly composed of carbolate of lime.

X. O. DUST.

[No. 176.]

Composition of X. O. Dust.

	Per cent.
Moisture	6.98
Organic and volatile matter	53.69
Carbon dioxid	3.40
Carbolic acid	Trace.

	Per cent.
Mineral matter	39.33
Sand	14.96
Ferric oxid and alumina	2.47
Calcium oxid	17.10
Magnesium oxid	2.09
Phosphorus pentoxid	.40
Potassium oxid	2.58

This mixture appears to be partly composed of carbonate of calcium with an excess of lime. No opinion as to the character of the organic and volatile matter can be based upon the data given, exclusive of the carbolic acid already mentioned.

ELECTRIC VERMIN EXTERMINATOR.

[No. 197.]

This insecticide is chiefly a mixture of calcium carbonate and calcium hydroxid with a very small amount of organic matter. Upon examination in this laboratory the organic matter present was found to consist of carbolic acid.

PAR' OIDIUM.

[No. 281.]

From the analyst's report this product would appear to consist of silicate of iron mixed with calcium sulphate. The analysis does not show the presence of sulphur and tobacco, both of which were found on examining a sample of the product in this laboratory.

ANTINONNIN.

[No. 282.]

A qualitative test of this substance showed that it contains dinitrocresols, together with soap and glycerin.

MISCELLANEOUS LIQUID INSECTICIDES AND FUNGICIDES.

BOLLENE INSECTICIDE.

Composition of samples Nos. 305, 644, 645.

[No. 305.]

	Per cent.
Carbon bisulphid	47.8
Mineral and essential oils	12.2
Water and residue	40.0

This analysis was made in accordance with the Texas law (see p. 61) regulating the sale of Paris Green and other insecticides and is good only until May 1, 1903.

[Nos. 644 and 645 (I & W).]

Of the two samples of this product received at the Bureau of Chemistry one consisted of a black oil (644), while the other (645) separated into two layers, one a black oil and the other a white milky emulsion. The first sample was fractionally distilled as it stood, and the second sample was separated into its two parts and each part subjected to fractional distillation.

No. 644 (I & W), results of distillation test.

	Per cent.
Below 95° C	[a] 51.07
95°–108° C	[b] .41
108°–130° C	[c] .57
130°–160° C	[c] .90
160°–190° C	[c] 2.31
190°–220° C	[c] 6.15
220°–260° C	[c] 3.69
260°–300° C	[c] 13.46
300°–350° C	[c] 6.16
350° C. +	[c] 1.02
Loss and residue	14.26

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[a] Practically all carbon bisulphid.
[b] Penetrating odor of some essential oil.
[c] Odor of petroleum products with paraffin which crystallizes out in the higher fractions.

No. 645 (I & W), results of distillation test.

WHITE EMULSION, 81.28 PER CENT.

	Per cent.
Below 95° C	[a] 1.24
95°–108° C	[b] 76.10
108°–130° C	[c] 13.47
Loss and residue	9.19

BLACK OIL, 18.72 PER CENT.

	Per cent.
Below 40° C	[d] 43.48
40°–65° C	[d] 22.00
65°–95° C	[d] 5.53
95°–108° C	[e] .50
108°–130° C	[e] 4.84
130°–160° C	[f] 8.21
160° C. +	[f] 6.53
Residue and loss	8.91

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[a] Odor of carbon bisulphid.
[b] Mostly water with solid particles of paraffin.
[c] Nearly all paraffin mixed with water.
[d] Nearly all carbon bisulphid.
[e] Some high-smelling essential oil present.
[f] Mineral oils with odor of petroleum products.

It is evident from the analyses of the two samples of bollene insecticide made in the laboratory that the first one (644 I & W) consists of about 50 per cent carbon bisulphid and 50 per cent crude petroleum with a very small quantity of some essential oil having a strong odor, while the second (645 I & W) consists of about 62 per cent water, 14 per cent carbon bisulphid, and 23 per cent petroleum, with a little essential oil and a small quantity of soap. Whether or not a compound of this composition, which is evidently very variable, would kill the bollworm is a matter for experimentation.

The results of the analyses of these two samples were forwarded to the manufacturer, who in reply said: "This analysis is not at all correct, and since the analysis was made I have perfected my preparation." Another letter was then sent, asking in what particular the analysis was unsatisfactory, but to this no reply was received. The analyses of these samples was repeated, but the same results were obtained. Besides this it is found that the analyses made in this laboratory gave very much the same results as those made by the Texas authorities from a qualitative standpoint. Unless the results obtained by the Texas authorities and by this laboratory are approximately correct the manufacturer is selling all of his goods under a false label, since the Texas law requires that all insecticides sold in the State shall comply with the official analysis on every package.

EXCELSIOR INSECTICIDE AND WOOD PRESERVER, RED CEDAR BRAND.

[No. 63, I & W.]

Results of distillation test.

	Per cent.
(1) 80°-110° C.	[a] 1.01
(2) 110°-180° C.	[a] .31
(3) 180°-230° C.	[b] 7.42
(4) 230°-270° C.	[b] 21.91
(5) 270°-300° C.	16.50
(6) 300°-330° C.	19.11
(7) 330°-355° C.	15.84
(8) Residue and loss	17.90

[a] Light oils with odor of benzene, toluene, etc.

[b] Give tests for phenol and cresols.

Fractions 4, 5, 6, and 7 are heavier than water. At about 340° C. the distillate begins to solidify in the condenser, and at 355° C. the condenser is so stopped up with naphthalene that it is necessary to cease distilling. This appears to be a sample of coal-tar creosote, and when properly used is of value both as an insecticide and a wood preserver.

FLY PREVENTATIVE.

[No. 202, I & W.]

Results of distillation test.

	Per cent.
(1) 80°-160° C	[a] 8
(2) 160°-215° C	[b] 7
(3) 215°-270° C	[c] 16
(4) 270°-350° C	[c] 19
(5) 350° C	[d] 33
(6) Residue and loss	[e] 17

[a] Light oil giving test for phenol and cresols.
[b] Heavier oil giving test for phenol and cresols.
[c] Thick oils.
[d] Thick oil with solid particles.
[e] Solid when cooling.

Fractions 1, 2, 3, 4, and 5 are all lighter than water. From the above analysis this sample appears to be a mixture of coal-tar creosote and petroleum. These substances are often used as fly preventatives.

KARBO KRESOLATE.

[No. 916, I & W.]

Results of distillation test.

	Per cent.
(1) 85°-105° C	[a] 26.4
(2) 105°-180° C	[b] 8.1
(3) 180°-220° C	[c] 7.6
(4) 220°-270° C	[d] 5.7
(5) 270°-285° C	[e] 8.8
(6) 285°-312° C	[e] 14.7
(7) Residue and loss	28.7

[a] Light oil and water mixed; smell of carbon bisulphid and benzine.
[b] Light oil, test for phenol and cresols.
[c] Oil, heavier than water, test for phenol and cresols.
[d] Oil heavier than water.
[e] Thick oil; pungent odor.

Fractions 3 and 4 contain solid naphthalene. A small amount of alkali is also present. From the distillation test it would appear that this sample consists of coal-tar creosote which has been boiled with resin and an alkali, or with resin, fatty matter, and alkali, to cause it to form an emulsion with water. From the size, appearance, and smell of the first fraction it also appears that some carbon bisulphid has been added. It is well known that creosote emulsions have given good results both as disinfectants and sheep dips. This preparation, therefore, would very likely be of value for the purposes intended.

PROFESSOR DE GRAFF'S CARPET BUG DESTROYER.

Composition of Professor De Graff's Carpet Bug Destroyer.

Constituents.	Sample No. 122.	Sample No. 129.
	Per cent.	*Per cent.*
Moisture	95.811	95.81
Residue from evaporation	4.189	....
Mercury	.782	.78
Chlorin	.265	.27
Sulphur trioxid	.484	.48
Alumina	.904	.90
Potassium oxid	.267	.26

These two analyses are given in two different reports of the Massachusetts State Experiment Station, but were very likely made upon the same samples, since they are identical.

The active poisonous principle of this solution is evidently mercuric chlorid, which is often used for the destruction of various insects which infest houses.

III. STATE LAWS GOVERNING THE COMPOSITION AND SALE OF INSECTICIDES.

A circular letter was sent to the various experiment stations asking that they forward copies of the laws in their respective States relative to the composition and sale of Paris Green and other insecticides. In cases where the experiment stations could not furnish the information a similar request was made of the secretary of state. It is believed that the following compilation of State laws is a complete résumé of all such laws passed in the United States.

CALIFORNIA.

AN ACT to prevent fraud in the sale of Paris Green used as an insecticide.

The people of the State of California, represented in senate and assembly, do enact as follows: It shall be the duty of each and every manufacturer of Paris Green (commercial aceto-arsenite of copper) to be used as an insecticide within this State, and of every dealer in original packages of said Paris Green manufactured outside of this State, before the said Paris Green is offered or exposed for sale, or sold within this State as an insecticide, to submit to the director of the California experiment station, at Berkeley, samples of said Paris Green, and a written or printed statement setting forth: First, the brand of said Paris Green to be sold, the number of pounds contained in each package in which it is put on the market for sale, the name or names of the manufacturers and the place of manufacturing the same; second, the statement shall set forth the amount of combined arsenic which the said Paris Green contains, and the statement so furnished shall be considered as constituting a guarantee to the purchaser that every package of such Paris Green contains not less than the amount of combined arsenic set forth in the statement.

SEC. 2. Every purchaser of said Paris Green in original packages which is manufactured outside of this State, who intends to sell or expose the same for sale, and every manufacturer of said Paris Green within this State, shall, after filing the statement above provided for with the director of the California agricultural station, at Berkeley, receive from the said director a certificate stating that he has complied with the foregoing statements, which certificate shall be furnished without charge therefor. Said certificate when furnished shall authorize the party when receiving the same to deal in this State in the said Paris Green. Any person who fails to comply with the terms of section 1 of this act shall not be entitled to such certificate and shall not be entitled to deal in said Paris Green within this State. Nothing in this section shall be construed as applying to retail dealers selling said Paris Green which has already been labeled and guaranteed.

SEC. 3. Paris Green, when sold, offered, or exposed for sale as an insecticide in this State shall contain at least 50 per centum of arsenious oxide and shall not contain more than 4 per centum of the same in the uncombined state.

SEC. 4. The director of the California State agricultural station, at Berkeley, shall examine or cause to be examined different brands of Paris Green sold, offered, or exposed for sale within this State, and cause samples of the same to be analyzed, and shall report results of analysis forthwith to the secretary of the State board of horticulture and to the party or parties submitting such samples, and such report shall be final as regards the quality.

SEC. 5. Any person or persons, firm, association, company, or corporation violating any of the provisions of this act, and any person who shall sell any package of Paris Green, or any part thereof, which has not been labeled as herein provided, shall be guilty of a misdemeanor, and shall be fined not less than $50 nor more than $200, together with cost of the suit, in an action caused to be brought by the State board of horticulture, through its secretary, in the name of the people of the State of California.

SEC. 6. The attorney-general of the State of California is charged with the prosecution of all such suits.

SEC. 7. This act will take effect immediately.—*Became a law by constitutional limitation February 28, 1901.*

LOUISIANA.

Act No. 131 of the general assembly of the State of Louisiana.

Be it enacted by the general assembly of the State of Louisiana, That the bureau of agriculture shall be charged with the duties of regulating the sale and purity of Paris Green as an insecticide in this State.

SEC. 2. That it shall be the duty of any manufacturer or dealer in original packages of Paris Green before the same is offered for sale in this State, to submit to the commissioner of agriculture a written or printed statement setting forth: First. The brand of Paris Green to be sold, the number of pounds contained in each package in which it is to be put upon the market for sale, and the name or names of the manufacturers and the place of manufacture. Second. The statement setting forth the amount of arsenic which they are willing to guarantee the said Paris Green to contain, and the statement so furnished shall be considered as constituting a guarantee to the purchaser that every package of said Paris Green contained not less than the amount of arsenic set forth in the statement.

SEC. 3. That every purchaser proposing to deal in Paris Green shall, after filing the statement above provided for with the commissioner of agriculture, receive from the said commissioner of agriculture a certificate stating that he has complied with the foregoing section, which certificate shall be furnished by the commissioner without any charge therefor; that the said certificate when furnished shall authorize the party receiving the same to deal in this State in Paris Green; that no person who has failed to file the statement aforesaid and to receive the certificate of authority aforesaid shall be authorized to deal in this State in Paris Green, and any person so dealing in this State without having filed the aforesaid statement and received the certificate aforesaid shall be liable for each violation to a fine not exceeding $250, which fine shall be recoverable before any court of competent jurisdiction at the suit of the commissioner of agriculture or of any citizen, and shall be disposed of as hereinafter provided; provided, further, that nothing in this section shall be construed as preventing the sale by retail dealers throughout the State of Paris Green which has already been guaranteed and labeled as provided for in this act.

SEC. 4. That it shall be the duty of the board of agriculture, or its commissioner, at the opening of each season, to issue and distribute circulars setting forth the brands of Paris Green, their percentages of arsenic as claimed by the dealers, and to more particularly describe them they shall be separated into two classes, viz: First, those brands containing 50 per cent or more of arsenic shall be classed as "strictly pure," and, second, all falling below this percentage shall be classed as "impure."

SEC. 5. That it shall be the duty of the commissioner of agriculture to cause to be prepared labels of suitable material, fitted to be attached to packages of Paris Green and to have printed thereon "guaranteed," with a blank space into which there may be stamped by the commissioner of agriculture the words "strictly pure," or "impure," as the guarantee may require; also the year or season in which it is to be used and a facsimile of the signature of said commissioner. The said labels shall be furnished by the said commissioner, to any dealer in Paris Green who shall have complied with the foregoing provisions of this act, upon the payment by said dealer to said commissioner of fifty cents for a sufficient number to label one hundred pounds of Paris Green.

SEC. 6. That it shall be the duty of every person, before offering for sale any Paris Green as an insecticide in this State, to attach or cause to be attached to each package one of the labels hereinbefore described, designating the quantity of Paris Green in the package to which it is attached. Any person who shall sell any packages of Paris Green, or any part thereof, which has not been labeled as herein provided for shall be guilty of a misdemeanor, and on conviction thereof shall be fined in the sum of one hundred dollars for each omission, which penalty may be sued for either by the Commissioner of Agriculture or any person for the uses hereinafter declared. Any person who shall counterfeit or use a counterfeit label prescribed by this act, or who shall use them a second time, shall be guilty of a misdemeanor, and on conviction thereof shall be fined in a sum not exceeding two hundred and fifty dollars, one-half of which shall be paid to the informer, which may be doubled or trebled at each second or third conviction, and so on progressively for subsequent convictions.

SEC. 7. That it shall be the duty of every person who sells a package of Paris Green, upon the request of the purchaser, to draw from the same and in the presence of the purchaser or his agent a fair or correct sample and to have the same securely enclosed and sealed and sent to the Commissioner of Agriculture for analysis by the official chemist of the State, and if upon analysis the said Paris Green shall be found below the guarantee given to the Commissioner of Agriculture and printed on the package, then the said seller shall be liable to the said purchaser for all damage accruing from said difference, recoverable in any court of competent jurisdiction in the State.

SEC. 8. That the copy of the official chemist's analysis of any Paris Green certified to by him shall be admissible as evidence in any court of the State on trial of any issue involving the merits of said Paris Green.

SEC. 9. That the Bureau of Agriculture shall adopt needful rules and regulations providing for the collecting of money arising from the sale of labels, or for any fines imposed under this act, and shall deposit the same with the Treasurer of the State.

NEW YORK.

AN ACT to amend the agricultural law, to prevent fraud in the sale of Paris Green.

The People of the State of New York, represented in Senate and Assembly, do enact as follows: Chapter three hundred and thirty-eight of the laws of eighteen hundred and ninety-three entitled "An act in relation to agriculture, constituting articles one, two, three, four, and five, of chapter thirty-three of the general laws," is hereby amended by adding a new article to be known as article eight and to read as follows:

SEC. 110. *State manufacturer and the dealer in original packages to file certificate with commissioner of agriculture.* After the passage of this act it shall be the duty of each and every manufacturer of Paris green within this state, and of every dealer in original packages of Paris green manufactured outside of this state, before the said Paris green is offered or exposed for sale or sold within this state, to submit to the commissioner of agriculture a written or printed statement setting forth, first, the brands of Paris green to be sold, the number of pounds contained in each package in which it is put upon the market for sale, the name or names of the manufacturers and the place of manufacturing the same; second, the statement shall set forth the amount of arsenic which the said Paris green contains, and the statement so furnished shall be considered as constituting a guarantee to the purchaser that every package of such Paris green contains not less than the amount of arsenic set forth in the statement.

SEC. 111. *Certificate to be given by the commissioner of agriculture to state manufacturer and dealer in original packages.* Every purchaser of Paris green in original packages, which is manufactured outside of this state, who intends to sell or expose the same for sale, and every manufacturer of Paris green within this State, shall, after filing the statement above provided for with the commissioner of agriculture, receive from the said commissioner of agriculture a certificate stating that he has complied with the foregoing statement, which certificate shall be furnished without any charge therefor; said certificate when furnished shall authorize the party receiving the same to deal in this state in Paris green. Any person who fails to file the statement aforesaid shall not be entitled to such certificate and shall not be entitled to deal in Paris green within this State; nothing in this section shall be construed as applying to retail dealers.

SEC. 112. *Composition of Paris green or analogous products.* Paris green or any product analogous to it, when sold, offered or exposed for sale, as such, in this state, shall contain at least fifty per centum of arsenious oxide. (*See Amendment.*)

SEC. 113. *Paris green to be analyzed at experiment station.* The director of the New York State agricultural experiment station, at Geneva, shall, under the direction of the commissioner of agriculture, examine, or cause to be examined, the different brands of Paris green sold, offered, or exposed for sale within the State, and cause samples of the same to be analyzed, and shall report the result of the analysis forthwith to the commissioner of agriculture.

SEC. 114. *Penalty for violations.* Any person or persons, firm, association, company, or corporation violating any of the provisions of this act shall be guilty of a misdemeanor, and shall be fined not less than fifty dollars nor more than two hundred dollars; and in addition thereto shall forfeit and pay unto the people of the state of New York the sum of one hundred dollars, together with the costs of the suit in an action caused to be brought by the commissioner of agriculture in the name of the people of the state of New York, as provided by section eight of the agricultural law.

2. This act shall take effect immediately. *Became a law March 23, 1898, by the approval of the Governor.* (*Laws of New York, ch. 113.*)

AMENDMENT TO PARIS-GREEN LAW.

In accordance with the suggestion made by the New York State Experiment Station in 1900, that portion of the Paris Green law which related to the definition of Paris Green was changed. The essential portion of the amended law embodying this change is as follows:

SEC. 112. *Composition of Paris green or analogous products.* Paris green, or any product analogous to it, when sold, offered, or exposed for sale as such in this state, shall comply with the following requirements:

First. It shall contain arsenic in combination with copper, equivalent to not less than fifty per centum arsenious oxide.

Second. It shall not contain arsenic in water-soluble forms equivalent to more than three and one-half per centum of arsenious oxide.

OREGON.

Only those sections of the act named below which deal directly with the subject of insecticides are given.

AN ACT to protect the fruit and hop industry of Oregon.

SEC. 3. It shall be unlawful for any person, firm or corporation doing business in the State of Oregon to sell paris green, arsenic, london purple, sulphur or any spray material or compound for spraying purposes, in quantities exceeding one pound, without providing with each package sold a certificate duly signed by the seller thereof guaranteeing the quality and per cent of purity of said materials.

SEC. 4. Any person, firm or corporation selling any of the above materials which do not conform with the certificate furnished therewith shall be deemed guilty of a misdemeanor, and upon conviction thereof shall be subject to a fine of not less than twenty-five ($25) dollars nor more than one hundred ($100) dollars.

SEC. 8. It shall be the duty of the Commissioner of the State Board of Horticulture of the district in which a violation of this act occurs to present the evidence of the case to the district attorney, whose duty it shall be to prosecute any person guilty of a violation of this act, which prosecution may be brought in any of the justice courts of this state.

SEC. 9. Inasmuch as the horticultural interests of the state demand immediate attention, this act shall be in full force and effect from and after its approval by the Governor. *Approved by the Governor, February 17, 1899.*

TEXAS.

AN ACT for the better protection of the farmer in the purchase of commercial fertilizers and commercial poisons used for destroying bollworms and other pests.

Be it enacted by the Legislature of the State of Texas: Before any commercial fertilizer or commercial poison, or any chemical or mixture used as a commercial fertilizer or commercial poison, such as London purple, arsenic, Paris green, or any poison used for the purpose of destroying the boll worm or other pests, is sold or offered for sale in this State, the manufacturer, agent, importer or party who sells or offers it for sale within this State shall deposit with the professor of chemistry of the Agricultural and Mechanical College a sealed tin can, bottle or jar containing not less than one pound of the fertilizer or commercial poison offered for sale; with an affidavit that it is a fair sample taken from several barrels, boxes, sacks, or from quantities in larger bulk, of the article thus to be sold or offered for sale; provided the unmixed substances, cotton seed meal, land plaster, salt, ashes, lime, green sand marl, uncrushed bones and animal excrements shall be exempt from the operation of the law.

SEC. 2. The manufacturer, importer, vender or agent of any commercial fertilizer or commercial poison, as referred to in Section 1 of this chapter, shall pay annually to the treasurer of the Agricultural and Mechanical College an analysis fee of fifteen dollars for each and every fertilizer or commercial poison sold, exposed or offered for sale within this State. Such payment shall be made at the time the sample of fertilizer or commercial poison is submitted to the professor of chemistry for analysis.

SEC. 3. After the analysis fee has been paid, as provided for in Section 2 of this chapter, it shall be the duty of the professor of chemistry of the Agricultural and Mechanical College to analyze or to have analyzed under his direction, any sample of a commercial fertilizer or commercial poison, in accordance with the requirements of the foregoing sections of the law. The professor of chemistry shall print the result of such analysis in the form of a label, which shall set forth the name of the manufacturer, the brand of such fertilizer or commercial poison, and the essential ingredients contained in such fertilizer or commercial poison, viz:

1. Available nitrogen and its equivalent in ammonia.
2. Soluble phosphoric acid; total available phosphoric acid.
3. Reverted phosphoric acid.
4. Total phosphoric acid.
5. Potash soluble in water.

This, however, shall not preclude the professor of chemistry from setting forth any other ingredient which the fertilizer may contain. And he shall place upon each label the money value of such fertilizer or commercial poison, computed from its composition as he may determine. He shall furnish such labels in quantities of 500 or multiple thereof at a cost of one dollar per hundred; the money to be paid directly to the treasurer of the Agricultural and Mechanical College.

SEC. 4. Every box, barrel, keg, or other package or quantity of commercial fertilizer or commercial poison (within the limitations of Section 1 of this law), in any shape or form, sold or offered for sale in this State, shall have attached to it in a conspicuous place the label as provided for in Section 3 of this law, with the signature of the professor of chemistry attached.

SEC. 5. The professor of chemistry, or any duly authorized agent of his, is hereby authorized to select from any package of commercial fertilizer or commercial poison sold, or exposed for sale in this State, a quantity not to exceed two pounds, for a sample to be used for the purpose of an official analysis and for comparison with the sample furnished by the manufacturer, agent, or vendor, for official analysis.

SEC. 6. Any manufacturer, agent, or vendor of any commercial fertilizer or commercial poison who shall offer or expose for sale any such fertilizer or commercial poison without having previously complied with the provisions of this chapter shall be fined not less than fifty and not more than five hundred dollars for each violation or evasion of this law.

SEC. 7. Any agriculturist or farmer, a purchaser of any commercial fertilizer or commercial poison in this State, may take a sample of the same under rules and regulations to be prescribed by the professor of chemistry of the Agricultural and Mechanical College and forward the same to him for analysis, which analysis shall be made free of charge.

SEC. 8. The revenues accruing from analysis fees and sale of labels, as provided for in this chapter, shall be expended by the board of directors of the Agricultural and Mechanical College for the maintenance of the chemical department and for such other purposes as they may determine.

SEC. 9. A copy of the official analysis of any fertilizer or commercial poison, or chemical certified to by the professor of chemistry, shall be admissible as evidence in any court of this State on the trial of any issue involving the merits of said fertilizer or commercial poison.

SEC. 10. Whereas the fact that there is no law authorizing the analysis of commercial fertilizers and commercial poisons, or the testing of adulterated poisons when used for the purpose of destroying the boll worm and other pests, and the better protection of the farmer, creates an emergency, and an imperative public necessity that the constitutional rule requiring bills to be read on three several days be suspended and that this act take effect and be in force from and after its passage, and said rule is so suspended and it is so enacted. *Passed by the Twenty-sixth Legislature of Texas, 1899.*

WASHINGTON.

AN ACT to provide against the adulteration of Paris Green and other compounds used for spraying trees and plants.

Be it enacted by the Legislature of the State of Washington, That it shall hereafter be unlawful for any person, firm, or corporation doing business in the State of Washington to sell or offer for sale adulterated or low grade Paris green, arsenic, London purple, sulphur, or any spray material or compound for spraying purposes.

SEC. 2. For the purposes of this act Paris green shall contain not less than fifty per cent of arsenic trioxide in combination, and not more than four per cent of water-soluble arsenic trioxide, and commercial arsenic shall contain not less than ninety-six (96) per cent of arsenic trioxide.

SEC. 3. Any person, firm, or corporation violating any of the provisions of this act shall be deemed guilty of a misdemeanor, and upon conviction thereof shall be punished by a fine of not less than twenty-five dollars nor more than one hundred dollars. All fines imposed for violation of the provisions of this act shall be paid to the treasurer of the county wherein the violation was committed and placed to the general fund of such county.

SEC. 4. That the State Commissioner of Horticulture and the county fruit inspectors under his supervision be charged with the enforcement of this act, with the assistance of the prosecuting attorney.

SEC. 5. It shall be the duty of the chemist of the State Agricultural Experiment Station to correctly analyze, without extra compensation and without extra charge to the state other than the necessary expenses, all substances and compounds used or offered for sale for spraying trees and plants, that the State Commissioner of Horticulture may send for analysis, and report to him without unnecessary delay the result of any analysis so made; any such chemist shall assist him in prosecuting violations of the law, by giving testimony, expert or otherwise. *Approved by the Governor February 26, 1901.*

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U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 77

H. W. WILEY, Chief of Bureau.

OLIVE OIL AND ITS SUBSTITUTES.

BY

L. M. TOLMAN and L. S. MUNSON.

Prepared under the direction of
W. D. BIGELOW,
Chief of Food Laboratory.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1903.

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LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., July 3, 1903.

Sir: I have the honor to transmit herewith for your inspection and approval a manuscript relating to olive oils and their adulterations, and embodying the investigations made in this Bureau of American and imported pure olive oils and adulterations thereof. This work was done under the act of Congress authorizing the Secretary of Agriculture "to investigate the adulteration of foods, drugs, and liquors," and I recommend that the results as reported herein be published as Bulletin No. 77 of the Bureau of Chemistry.

Respectfully,

H. W. Wiley,
Chief of Bureau.

Hon. James Wilson,
Secretary of Agriculture.

INTRODUCTION.

The work reported in this bulletin was begun on imported oils received through the customs officers, with a view to determining the nature of the products brought into the United States from abroad and sold as olive oil. It was then thought advisable to go into the open market and secure oils sold in retail stores under foreign labels in order to make a comparison of olive oil as it is received by the customs officers and as it is sold to the consumer. Having thus collected considerable data on oils of other countries, it seemed expedient to make a study of American olive oils for the sake of comparison. Many samples were therefore purchased in the open market, and a number of representative manufacturers were requested to furnish samples of their product as it left the mill. These requests were quite generally complied with, and in each case the manufacturer sent with his sample an affidavit as to its purity. Employees of the Bureau visited New York, Philadelphia, Washington, Boston, New Haven, Rochester, Buffalo, Burlington, Vt., New Orleans, Chicago, St. Louis, and San Francisco, and purchased both American and foreign oils found in as many stores, selected at random, as it seemed advisable to visit.

The relatively low price at which imported oils are sold as compared with California oils may often be attributed to their inferior quality. In selecting material for the work reported in this bulletin no samples were taken which were not sold as olive oil. Oils sold under other names, or simply as salad oil, were not included. It is a matter especially worthy of comment that the California oils bought in the open market were all of superior quality, and that only two of the fifteen samples so obtained contained any oil other than olive oil. Of these two samples one was not labeled with the name of the manufacturer, but instead bore the name of the dealer.

Following the usual custom in such matters, all manufacturers resident in the United States were notified of the results of the examination previous to their publication, and no protests were received except those relating to the two oils mentioned. The dealer from whom one of these samples was obtained replied that he supposed that he was handling a high-grade article, while the manufacturers of the other sample wrote us that they had ceased to make olive oil because, while making a high-grade article, they could not compete with inferior goods which were sold under the labels of first-class olive oil, and had decided to discontinue its manufacture. They stated that they employed no cotton-seed oil or other substitute at any time. Dealers whose names appeared on the labels as importers or manufacturers' agents were also notified.

For the lack of a better term, a number of salad oils are referred to in this bulletin as "olive oil substitutes." By this it is not intended to class them as inferior in any sense, or to disparage their legitimate use or their sale when labeled in such a way as to notify the purchaser of their nature. They are included in this bulletin, however, merely because, being cheaper oils, they are sometimes fraudulently substituted for olive oil and sold either at a price above their relative value, or at a price which brings olive oil into unfair competition. Such practices are fraudulent, and it is highly important for those engaged in the manufacture of olive oil, which has become an important industry in California, that they be forbidden.

Two Federal laws were passed by the last Congress which have a bearing on this subject. One of these (Public—No. 223) forbids the misbranding or false labeling of food products as to the place of their manufacture. The other (Public—No. 158) prohibits the importation into the United States of foods which are "found to be dangerous to health, or which are forbidden to be sold or restricted in sale in countries in which they are made or from which they are exported, or which shall be falsely labeled in any respect in regard to the place of manufacture or the contents of the package."

With these laws on the statute books it becomes important that purchasers should carefully read the labels on their purchases and note that the name of the manufacturer and the place of manufacture are distinctly stated. It is altogether probable that in the future oils distinctly labeled will be found to be as represented, as ordinarily fraudulent manufacturers will attempt to evade the law by omitting the place of manufacture rather than violate the law by making a false statement regarding it. As an illustration of this a label is reproduced (Pl. I, fig. 1) which was found on a bottle of maize oil purchased as olive oil. This label does not distinctly state that the bottle contains olive oil, but it is worded in such a way that it is only upon close examination that the purchaser will note the fact that the oil is represented not as "superior olive oil," but as "superior to any olive oil."

Among other instances of misbranded oil are a number which bear French or Italian labels and contain cotton-seed oil. As illustrations of this two labels are reproduced (Pl. I, fig. 2, and Pl. II, fig. 2) which are believed to have originated in this country.[a] The prevalence of the sale of cotton-seed oil under labels of this nature has led to frequent rumors that the cotton-seed oil exported from the United States is largely used as an adulterant or substitute for olive oil, and is sent back to us under another label and at greatly increased prices. It is difficult to understand how this idea could have gained credence. The shipment of the oil twice across the ocean would seem a needless expense. Oils of American origin are never subjected to a more rigid examination than imported oils. In view of these facts we would expect even foreign manufacturers to establish mixing and bottling establishments here, if they desire to handle American oil, and confine their importation to the labels.

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[a] See page 56.

In the following pages several instances of the presence of cheaper oils imported as olive oil are noted, but not a single instance has been found of the use of cotton-seed oil as an adulterant or substitute for olive oil in goods known to be imported,[a] although samples of imported olive oil were obtained from the customs officers of several of our leading ports, and every effort was made to secure a fair representation of the oils imported. We are therefore brought to the conclusion that cotton-seed oil is not imported under olive oil labels, as is frequently stated. Since the completion of our work on this subject this idea has been confirmed by a report from Consul-General R. P. Skinner, as a result of his investigation of the manufacture of olive oil in France. Mr. Skinner calls attention to the fact that edible olive oil to the value of $1,200,000 is brought annually into the United States, and that Marseilles alone has sometimes imported $3,000,000 worth of cotton oil in a single year. Mr. Skinner then says: "Therefore, if we also assume, a most improbable hypothesis, that these oils contain 25 per cent of cotton oil, we have accounted for cotton oil to the value of merely $300,000, a mere drop in the bucket as compared with the value of our cotton-oil exports." Another form of fraud is illustrated in Plate II, fig. 1, which is a reproduction of a label on a bottle received from the appraiser's stores imported as olive oil, but containing peanut oil.

I take this opportunity to express our indebtedness to all the American manufacturers who have cooperated in this work by furnishing the laboratory with samples of their oil, accompanied by affidavits as to their purity and descriptions of their methods of manufacture; and also to V. Villavecchia, custom-house chemist, Rome; Giacomo Dellepiane fu Andrea, Genoa; G. E. Colby and W. C. Blasdale, of the University of California, Berkeley, Cal., who have supplied samples of olive oil of known purity from different regions, and the V. D. Anderson Company, who prepared several samples of olive-oil substitutes for our laboratory.

W. D. BIGELOW,
Chief of Food Laboratory.

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[a] Two samples recently received from the custom-house have contained cotton-seed oil.

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II. Fig. 1.—Label on olive oil mixed with peanut oil. Fig. 2.—Label on bottle of cottonseed oil 56

OLIVE OIL AND ITS SUBSTITUTES.

GENERAL DISCUSSION.

STATISTICS OF THE OLIVE OIL TRADE.

Nearly the entire amount of olive oil used in this country comes from Italy, France, and California. The average annual total importation for the ten years next preceding 1901 was 803,716 gallons. Of this amount France sent us annually an average of 257,586 gallons and Italy 498,493 gallons. There are no means of ascertaining the amount produced in California, but it is probably a great deal less than the amount imported, as the California oil is scarcely known in the eastern markets, and is by no means the only oil offered on the western markets. Evidence of this is the fact that of 68 samples of commercial oils bought outside of California but two were California oils. On the other hand, of 29 samples of commercial oils bought in California 13 were produced in California, 4 were French oils, and 12 were Italian oils.

In 1897 Elwood Cooper[a] reported 2,500,000 olive trees in California capable of producing, besides dried and pickled olives, 2,000,000 gallons of oil. This amount, however, is greatly in excess of the present production, owing to the fact that, as shown later, both the French and Italian oils can be imported cheaper than the actual cost of production of the California product. The above figures are of interest in showing that California is capable of producing the entire amount of olive oil consumed in this country.

TABLE I.—*Importations and valuations of olive oil.*

Year.	Importations.				Valuation per gallon.			Total valuation of oils imported.
	From France.	From Italy.	From all other sources.	Total importations.	French oils.	Italian oils.	Average for all oils imported.	
	Gallons.	*Gallons.*	*Gallons.*	*Gallons.*				
1891	217,628	326,748	61,133	605,509	$1.59	$0.99	$1.21	$733,489
1892	222,534	431,322	52,630	706,486	1.72	.97	1.24	876,613
1893	261,332	379,150	46,370	686,852	1.69	1.01	1.29	891,424
1894	205,089	506,647	45,742	757,478	1.74	.97	1.20	909,897
1895	261,695	461,215	52,136	775,046	1.67	.96	1.22	952,405
1896	278,791	610,332	53,475	942,598	1.70	.92	1.17	1,107,049
1897	328,943	558,115	46,509	928,567	1.61	.98	1.22	1,134,077
1898	243,874	457,939	35,064	736,877	1.69	1.01	1.25	923,804
1899	280,515	609,038	40,489	930,042	1.64	.94	1.17	1,090,250
1900	275,461	649,423	42,818	967,702	1.65	1.00	1.21	1,170,871

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[a] Address before the State Fruit Growers' Association of California.

Table I, prepared from the report of the Bureau of Statistics, Treasury Department, for 1900 (p. 289), gives the importations of edible olive oils, which pay a duty of 50 cents per gallon, and the valuation of these oils. According to this table the Italian oil is much cheaper than the French product, the average difference in price being about 60 cents per gallon. This difference is partly due to the packages in which the oils are shipped. The Italian oils are shipped largely in bulk or in tin cans, while the French oils are nearly always bottled and labeled in France. Of 27 samples of Italian oils furnished by the Treasury Department for examination, one was in bottle, one in tin, and the remainder in bulk. Of 27 samples of French oils from the same source, 17 were in labeled bottles and 10 were in bulk. Another reason for the lower average price of Italian oils is that a large amount of low-grade oil is imported. The price of the high-grade oils from both countries is about the same.

Table I shows that Italian oils are valued at about $1 per gallon and French oils at $1.60; adding 50 cents per gallon for duty to each of these would make their values $1.50 and $2.10, respectively. According to B. M. Lelong[a] the cost of production of California oil is $1.88 per gallon; adding to this 50 cents per gallon for bottling would make the actual cost of preparing the oils for market $2.38.

Table II gives the retail price per gallon of 83 samples of commercial oils, of which 67 are unadulterated and 16 adulterated. The figures on the unadulterated oils are in much the same ratio as the prices of the imported oils given in Table I and the actual cost of manufacture of the California oils as stated above.

TABLE II.—*Retail price per gallon of commercial samples.*

	Unadulterated oils.			Adulterated oils.		
	California.	French.	Italian.	California.	French.	Italian.
Number of samples	9	30	28	2	6	8
Average price per gallon	$5.87	$4.92	$4.20	$3.58	$4.03	$2.43
Maximum price per gallon	6.65	6.65	8.19	3.84	6.58	3.32
Minimum price per gallon	4.86	2.56	1.79	3.32	1.66	1.79

These figures show that California oils retail at a slightly higher price than French oils and French oils at a slightly higher figure than Italian oils. Referring to the price of adulterated oils, it will be seen that price is not always, or even generally, an indication of purity. The California oils show very little variation in price. This is due to the fact that all of the oil is of the highest quality. With both French and Italian oils some very low grades are placed on the market. The best grades of all three varieties of oil bring about the same price.

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[a] Report of the Secretary of the California State Board of Agriculture, 1897.

MANUFACTURE OF OLIVE OIL.

The processes of manufacturing oil from the olive are very simple. The following general description is taken from a report to the governor of California by the State board of horticulture. The fruit is picked when ripe and dried either in the sun or by means of dryers. It is then crushed in a mill and the oil extracted by a powerful press, the details of these processes varying in different mills. In some places the olives are mixed with hot water before making even the first pressing. The first oil coming from the press is called the "virgin oil" and is the highest grade of salad oil. The pulp left from the first operation is subjected to a much higher pressure to produce the second-pressing oil. As a rule, this is mixed with the "virgin oil." The pulp is now treated with hot water and pressed again, when a third-grade oil is produced which is used for the table and also for lubricating fine machinery and for burning. The pulp still contains oily matter which is extracted with carbon disulphid, and used in making castile soap and in dyeing. In this country very little of this lower-grade product is made. The oil from the different pressings is run into tanks with water and allowed to stand until the pulp and gummy matters settle out. The oil is drawn off carefully, filtered, and stored in a cool, dark place until ready to be bottled, as olive oil becomes rancid very quickly if exposed to light and heat.

The following letters from California manufacturers of olive oil give in greater detail the processes used at the various mills:

SANTA CLARA, CAL., *May 7, 1902.*

My olives are picked when the average is ripe, but not *dead* ripe. They are spread on trays and allowed to evaporate a portion of the "water of vegetation" before pressing. They are crushed in an old-fashioned crusher, a revolving granite wheel worked by horsepower, and pressed in Italian oil sacks (bruscole), which are made of a kind of rush (guinco marino), which I import for the purpose. Practically all the work is done by "rule of thumb;" i. e., by careful study of the condition of the olives, etc. We try to keep the temperature of the pressing room at about 70° F., but the olives are not heated and come in cold. The run of a pressure is a mixture of water and oil, fine material of the olive, and oil. After the run has stood a short time the oil has in part come to the surface and the water sunk to the bottom with an intermediate stratum of a mixture of oil, water, and fine material. The oil is skimmed off the surface as soon as it appears in sufficient quantity, and this skimming is continued indefinitely until the oil is called for and filtered for bottling. The processes are the old ones in use in all the old countries where oil is expressed, and, so far as I can judge, the best known up to the present time. There is absolutely no secret. All depends on an intimate knowledge of the olive and these processes, and I employ selected Italians from the oil districts of Tuscany to secure efficient work. Frequent experiments are tried here in various lines, hoping to lessen the labor, but so far without any serious success. The De Laval Company tried a separator here this winter, and we hope that the expert, now fully acquainted with the difficulties to be met, may work out an oil separator or a series of separators which will take the olive just picked and give the refined oil at the end of the process or processes. An exceedingly able English mechanic has tried two experiments with a new press this season here, and has since continued his experiments with olives sent to San Francisco, of which I have not as yet learned the result. I am confident that we shall before long have a machine which will avoid the costly process of partial evaporation and handle the olives as they come from the tree without sacks or the cider-press outfit, both of which mean much labor and expense.

Yours, very truly, EDWARD E. GOODRICH.

JAMACHA P. O., SAN DIEGO COUNTY, CAL., *March 30, 1902.*

Yours of March 22, asking for information in regard to olive oil, just received.

I have no secret process. I inclose a printed description of my process, which very clearly shows our method. We use no heat at any stage of the process. This cold-water separator I consider a great improvement over the old way of storing in large settling vats and skimming off the oil as it rises. By this process the oil is separated from the lees as fast as pressed. Every morning the oil expressed the day before is taken from the separator and placed in the racking tanks. I use only ripe olives and press them fresh from the tree. I put up two grades, only one of which we bottle. No. 2 is sold only in bulk to our local trade.

The cost of production of course depends entirely upon the price of olives. This season we have paid $40 per ton; this is $30 less than we have ever paid before. At this price for olives I figure my oil costs me very close to $1.50 per gallon for the season's run. Our olives in this locality are nearly all Mission. What few we have of others we mix with them.

Yours, very truly, C. M. GIFFORD.

C. M. GIFFORD'S PROCESS OF EXPRESSING OLIVE OIL.

My process of expressing olive oil is very simple and is as follows:

Only ripe olives are used. The fruit is first crushed in a large stone basin by means of a revolving stone wheel; it is then taken to the presses, where the oil and juice are separated from the pomace. The oil and juice are then passed through our new cold-water separator, where it comes in contact with a stream of pure spring water under heavy pressure, which thoroughly washes and removes from the oil all vegetable matter. Next it goes to the racking tanks, where it is thoroughly racked and allowed to clarify. It is now taken to the filter room and filtered through paper, made for this purpose, directly into the bottles.

The utmost care and cleanliness are absolutely necessary in the making of a high-grade olive oil, the product being very susceptible to taint.

SANTA BARBARA, CAL., *November 23, 1901.*

In my process the fruit is first dried in the sun for about two or three weeks, then crushed, pits and all, to a fine pulp, mixed with hot water (nearer 200° than 90° F., contrary to the accepted theory), and pressed through scalded Russia crash. This product is then stored for some months in heavy, *very clean* whisky barrels with the air entirely excluded, each barrel having considerable of the black, watery fluid, or lees, as it is called here, into which all impurities settle from the oil. If it shows signs of fermentation when first stored, which occurs some years, I place a glass siphon in the top of the barrel, cement in a cork with paraffin, and allow the gas to escape in pure water. The oil is then drawn off from a point just above the lees and filtered through paper, a sample of which is inclosed herewith. I never crushed the pits, but have made up my mind that the flavor of the oil is not changed at all by doing so. This oil brings the highest price of any oil I know of ($15 a case of 12 bottles, 6 to the gallon), and yet I am convinced it has always cost me that much or more to produce it, although I charge up my fruit at 4 cents a pound, which is what we paid when we were running the El Montecito Manufacturing Company, but they are quoted now at just half ($40 a ton), which is just twice what I have to pay for picking (1 cent a pound). We have spent a small fortune, I think fully $50,000, in trying to introduce *pure olive oil*, and failed, closing the mill indefinitely. I think in our mill the oil cost us $1.17 a bottle, and much of it had to be sold at 40 cents.

Respectfully, yours, PEVERIL MEIGS.

METHODS OF ANALYSIS AND INTERPRETATION OF RESULTS.

The methods used in this work are not given in this bulletin if they are to be found in Bulletin 65[a] of this Bureau or are modifications of methods already published. More attention will be given to the discussion of the application of these methods and their limitations in the detection of various adulterants. In many control laboratories it is customary to make a few of the simpler determinations and qualitative tests, and to judge the state of purity of the oil from the results obtained by these determinations. While in many cases this is sufficient, especially if gross adulteration has been practiced, it is entirely inadequate in case the amount of foreign oil added is small. In such an instance a thorough examination is essential to properly judge an oil. The condition of an oil may frequently be such as to give abnormal values, even though it is absolutely pure. For example, an oil having a high acidity would give too low results for specific gravity, index of refraction, and possibly too low a Hübl value, especially if the free fatty acids had become oxidized. If the determination of acidity were made, it would explain at once the abnormal results. Frequently an oil that may be pronounced adulterated upon superficial examination will upon more careful analysis be found to be unadulterated.

The qualitative tests, while in many cases conclusive, must be used with caution. Halphen's test, if positive, is satisfactory proof of cotton-seed oil, but, as will be shown later, the heated oil does not respond with this reagent. The tests of Baudouin and of Villivecchia for sesame oil are conclusive if used with proper precautions, but some oils from Tunis give a color with this reagent that may easily be mistaken. The Bechi test for cotton-seed oil and the Renard test for peanut oil must be used with extreme care or misleading results will be obtained.

SPECIFIC GRAVITY.

The specific gravity was determined by the use of a hydrometer graduated from 0.9100 to 0.9300 at 15.5° C. and reading to 0.0002.

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[a] Provisional methods for the analysis of foods, adopted by the Association of Official Agricultural Chemists, November 14, 15, 16, 1901.

The readings were made as near 15.5° C. as possible and correction for temperature made by the following formula:[a]

$$G=G'+0.00063\ (T-15.5^\circ\ C.).$$

G = specific gravity at 15.5° C.

G′ = specific gravity at T.

0.00063 = mean correction for 1° C. (for olive oil).

The correction factors for other oils are given in the provisional methods for the analysis of foods.[b]

The determination of specific gravity is a quick way of detecting gross adulteration of olive oil with the seed oils, practically all of which have a higher specific gravity. The range of specific gravities on pure olive oils of the grade used for salad purposes is quite narrow. On 33 samples of pure California oils the specific gravity varied from 0.9162 to 0.9180, with an average of 0.9168. One sample, No. 673, had a specific gravity of 0.9149, but had 12.11 per cent of free acid, which explains the low gravity and excludes the oil from use for salad purposes. Eighteen samples of Italian oils gave a range of from 0.9155 to 0.9180, with an average of 0.9163. Blasdale[c] on 11 samples of California oils found a variation of from 0.9161 to 0.9174. Milliau[d] found on French oils a range of from 0.9169 to 0.9172, and for Tunis oils[e] a range of from 0.9170 to 0.9196, with an average on 49 samples of 0.9183. De Negri and Fabris[f] in a very exhaustive examination of Italian oils found on 70 samples a range of from 0.9160 to 0.9180, and an average of 0.9166. These authors found one sample of oil made from rotten olives which had a specific gravity of 0.9145. This low figure was most likely due to the presence of free fatty acids, which were not determined, but which were probably present in sufficient amounts to materially influence this constant and to exclude the product from edible oils. Colby[g] gives the range for California oils of 0.9140 to 0.9185, but in the absence of definite data regarding the amount of free acids present the lower limit can hardly be accepted for a normal oil. From the above figures it is apparent that there is but little variation in the specific gravity of edible olive oils from different sources and from different varieties of olives.

The specific gravity of California, French, and Italian olive oils varies from 0.9140 to 0.9185, while the oils from Algeria range as high as 0.9196. Zammit[a] called attention to the fact that the presence of free acids in olive oils reduces the specific gravity, and the following table taken from his results shows the effect of excessive amounts:

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[a] Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 33. Winton, Connecticut Agr. Expt. Sta. Rept., 1900, pt. 2, p. 149.

[b] U. S. Dept. Agr., Bureau of Chemistry Bul. 65, p. 21.

[c] Jour. Amer. Chem. Soc., 1895, **17**: 935.

[d] Bul. du Ministère de l'Agriculture, 1895, p. 139.

[e] Milliau, Bertainchand et Malet, Bul. de l'Agriculture et Commerce de Tunis, "Rapport sur les huiles d'olive de Tunisie, 1900."

[f] Annali del Lab. chim. centr. delle Gabelle, vol. 1, Gli Olii, pt. 1, p. 38.

[g] California Agr. Expt. Sta. Rept., 1897–98, p. 169.

TABLE III.—*Effect of excessive amounts of free acids in olive oils on their specific gravity.*

Free acid as oleic.	Specific gravity 15.5° C..	Free acid as oleic.	Specific gravity 15.5° C..
12 per cent	0.9147	25 per cent	0.9128
13 per cent	.9139	27 per cent	.9136
14 per cent	.9143	28 per cent	.9124
15 per cent	.9144	31 per cent	.9122
20 per cent	.9132		

Table IV gives the range of specific gravity of all the oils considered in this bulletin. Oils having an excessive amount of free acids, and consequently abnormal specific gravity, have been excluded from the ranges given in the column of "Results obtained in Bureau of Chemistry." This explains in many cases the lack of agreement between the results from the two sources. An example of this is one sample of peanut oil having 13.51 per cent of free acids and a specific gravity of 0.9155. The other three samples of peanut oil have specific gravities of 0.9186 and above, which are considered about normal for this oil.

TABLE IV.—*Specific gravity of oils.*

Kind of oil.	Results obtained in Bureau of Chemistry.	Compiled results.
California olive	0.9162 to 0.9180	0.9140 to 0.9185
Italian olive	.9155 to .9180	.9158 to .9180
French olive		.9169 to .9172
Algerian olive		.9170 to .9196
Lard	.9148 to .9175	.9150 to .9160
Rape	.9143 to .9163	.9112 to .9184
Mustard	.9147 to .9193	.9125 to .9183
Almond	.9186	.9154 to .9200
Peanut	.9186 to .9188	.9110 to .9220
Cotton-seed	.9226 to .9236	.9160 to .9362
Sesame	.9218	.9200 to .9250
Maize	.9233 to .9256	.9213 to .9255
Sunflower	.9201 to .9205	.9240 to .9262
Poppy	.9239 to .9244	.9240 to .9370

INDEX OF REFRACTION.

This determination was made with a Zeiss butyro-refractometer, the readings being made as nearly at 15.5° C. as possible. As it is not always convenient to make the readings at this temperature, the factor to be used for the correction for temperature was determined on oils likely to be used as substitutes for olive oil.[b] The butyro-refractometer, on account of its ease of manipulation, is very widely used, but its empirical scale, with divisions of varying value in refractive indices, requires for an accurate correction for temperature that the scale readings be calculated to indices of refraction and the temperature correction applied thereto. The reason for this can be seen from Table V.

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[a] Rev. Int. Fals. Alim., 1899, **12**: 84.

[b] Tolman and Munson, Jour. Amer. Chem. Soc., 1902, **24**: 754.

TABLE V.—*Value of butyro-refractometer degrees in refractive indices on different parts of the scale.*

Scale division.	N_d.	Difference.
0	1.4220	0.0080
10	1.4300	
40	1.4524	.0069
50	1.4593	
90	1.4840	.0055
100	1.4895	

From this it will be seen that ten scale divisions from 0 to 10 on this instrument equal 0.0080 in index of refraction, while from 90 to 100 they equal 0.0055. It is evident that if, as shown in Table VII, the change in the refractive index for 1° C. is practically a constant, then the change in the scale division of this instrument for 1° C. must be a variable and will increase as the scale reading increases. This is shown in Table VI, which gives the readings on rape oil. The change for 1° C. decreases as the temperature rises.

TABLE VI.—*Variable temperature correction in butyro-refractometer readings.*

Temperature.	Scale readings.	Change for 1° C.	
		In scale reading.	In refractive index.
	Degrees.	*Degree.*	
2.8° C.	82.0	0.60	0.000360
21.8° C.	70.5		
21.8° C.	70.5	.57	.000360
38.4° C.	61.0		

Table VII gives the determinations of the temperature corrections of various oils.

TABLE VII.—*Temperature corrections for various oils.*

Laboratory No.	Kind of oil.	First reading.		Second reading.		Third reading.		Increase for 1° C.		
		Temperature.	Butyro-refractometer reading.	Temperature.	Butyro-refractometer reading.	Temperature.	Butyro-refractometer reading.	Between first and second reading.	Between second and third reading.	In index of refraction.
		° C.	*Degrees.*	° C.	*Degrees.*	° C.	*Degrees.*	*Degree.*	*Degree.*	
23605	Olive	3.4	76.5	22.0	65.5	37.8	56.5	0.59	0.57	0.000366
506	.....do	3.2	76.0	22.2	64.8	36.8	56.5	.59	.57	.000366
23460	.....do	3.2	76.0	22.2	65.0	43.3	53.0	.58	.56	.000360
493	Poppy seed	9.0	82.5	19.9	75.6	41.0	63.2	.63	.59	.000369
491	Maize	2.8	84.5	21.8	73.0	39.6	62.5	.60	.59	.000366
498	Sunflower	3.0	81.0	21.8	69.5	39.0	59.5	.61	.58	.000369
23624	.....do	3.0	81.2	22.0	70.0	39.0	60.0	.59	.58	.000364
496	Rape	2.8	82.0	21.8	70.5	38.4	61.0	.60	.57	.000363
490	.....do	3.4	82.2	22.0	71.0	38.8	61.4	.60	.57	.000364
486	Mustard	2.8	84.5	22.0	72.5	38.0	63.2	.62	.58	.000360
495	Black mustard	3.2	84.5	22.0	73.0	39.4	63.0	.61	.57	.000361
487	Lard	3.0	74.5	22.0	63.4	37.6	54.6	.58	.56	.000363
22434	.....do	3.0	75.2	22.0	64.0	39.2	54.0	.59	.57	.000372
499	Peanut	3.0	78.5	22.0	67.2	37.2	58.5	.59	.57	.000365
23656	.....do	3.3	76.0	22.0	64.5	44.2	52.3	.61	.55	.000366
492	Peanut and cotton seed	3.4	82.0	22.0	70.5	42.5	58.8	.61	.57	.000369
489	Sesame	3.4	81.2	22.0	70.0	38.2	60.5	.60	.58	.000370
436	Cotton seed	3.4	81.5	22.2	70.0	37.6	61.2	.61	.57	.000368
	Average									.000365

From these results it is seen that the correction of the index of refraction for 1° C. is practically a constant for all of the oils examined, while the correction in scale divisions varies from 0.55 to 0.63 for 1° C. For great changes of temperature it is not accurate to use the scale division factor, but the results must be reduced to indices of refraction and the correction applied to them. The following formula may be used to correct for a range of 10 degrees:

$R = R' + X (T' - T)$.
R = reading corrected to T.
R' = reading at T'.
T = desired temperature.
T' = temperature at which readings are made.
X = change in scale division caused by a change of temperature of 1° C. ($X = 0.55$ for butters; 0.58 for oils that read from 60° to 70° on the scale; and 0.62 for oils that read from 70° to 80°.)

The determination of the index of refraction has much the same value in the detection of adulteration as the determination of specific gravity. The forty-two samples of California oils examined had a range of from 1.4703 to 1.4718, with an average of 1.4711. These limits exclude one sample having 44 per cent of free acid and an index of 1.4672, as free acid reduces the index of refraction of an oil. Blasdale[a] on eleven samples of California oils obtained a range of 1.4710 to 1.4716. Colby[b] gives a range for California oils of from 1.4689 to 1.4717, but this is probably too wide for oils which can be classed as salad oils. On eighteen samples of pure Italian oils reported in this bulletin there was found a range of from 1.4703 to 1.4713. There is therefore a total variation in the determinations made in this laboratory of from 1.4703 to 1.4713, with no difference between the California and Italian oils. Table VIII gives the index of refraction obtained on various oils.

TABLE VIII.—*Butyro-refractometer readings and refractive indices of oils.*

Kind of oil.	Number of samples.	Butyro-refractometer readings at 15.5° C.	Index of refraction at 15.5° C.
		Degrees.	
California olive	42	66.9 to 69.2	1.4703 to 1.4718
Italian olive	18	67.3 to 68.5	1.4706 to 1.4713
Lard	4	68.8 to 69.5	1.4702 to 1.4720
Almond	1	70.9	1.4728
Peanut	3	70.0 to 71.3	[c] 1.4723 to 1.4731
Mustard	5	74.5 to 76.5	1.4750 to 1.4762
Rape	4	74.1 to 74.8	1.4748 to 1.4752
Sesame	1	73.3	1.4742
Sunflower	1	72.7	1.4739
Maize	4	75.6 to 77.5	1.4757 to 1.4768
Cotton-seed	4	72.3 to 75.6	1.4737 to 1.4757
Poppy	1	77.8	1.4770

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[a] Jour. Amer. Chem. Soc., 1895, 17: 935.

[b] California Agri. Expt. Sta. Rept., 1897–98, p. 169.

[c] One sample of peanut oil containing 13.51 per cent of free acids had an index of refraction of 1.4707.

MAUMENÉ NUMBER[a] AND SPECIFIC TEMPERATURE REACTION.[b]

The method used in making this determination was that described in the Provisional Methods for the Analysis of Foods.[c]

The specific temperature number was calculated by the following formula:

$$S = \frac{100\ A}{B}.$$

S = Specific temperature number.
A = Maumené number.
B = The rise of temperature obtained with pure water under the same conditions as those used in the Maumené test.

This simple test if carried out properly is a most valuable and rapid means of judging the purity of olive oil. Lard oil is practically the only oil which would be used as a substitute that has as low a figure.

Different analysts have obtained widely varying figures for this test, but it is easily shown that these are due to the use of acids of varying strength. Colby,[d] in preparing standards for the Association of Official Agricultural Chemists, gives from published results a range for this test of 32 to 47; but such figures as these have no value as they represent results obtained by different methods and different strengths of acid. Blasdale[e] found on a number of California oils a range of 45 to 47. With the 42 samples of California oils of known purity examined in this laboratory a range of 43.5 to 52.1 was found, with an average of 46.8. In contrast to these results, Milliau,[f] in his report on the oils of Tunis, gives a range of 30 to 36. These figures represent the variation found in 56 samples of oils from varied sources and processes of manufacture. Milliau,[g] in an earlier report on the olive oils of Europe, gives a range for the Maumené figure of 30 to 35. This wide difference in results is due entirely to the strength of the acid used in making the test. The actual limits where the same method and the same strength of acid are used are quite narrow. In preparing acid for this test Milliau[h] diluted 95 parts by volume of pure 66° Baumé acid with 5 parts of distilled water. The acid prepared in this way gives a rise of temperature with water of about 38° C., and compares satisfactorily when calculated to the specific temperature number with results obtained by the use of strong acid giving a rise of temperature with water of 45 to 46° C. In this laboratory an acid with a specific gravity of 1.844 was used, which gave a rise of temperature with water of 45° C. Thus it can be seen that the Maumené numbers alone have no comparative value. Either the exact strength of the acid must be given, or, perhaps better, the rise of temperature with water under standard conditions.

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[a] Comp. Rend., 1852, **35**: 572.

[b] Thomson and Ballantyne, Jour. Soc. Chem. Ind. 1891, **10**: 233.

[c] U. S. Dept. Agr., Bureau of Chemistry Bul. 65, p. 31.

[d] California Expt. Sta. Rept., 1897–98, p. 169.

[e] Jour. Amer. Chem. Soc., 1895, **17**: 935.

[f] Milliau, Bertainchand and Malet, Rapport sur les huiles d'olive de Tunisie, 1900.

[g] Bul. du Ministère de l'Agriculture, 1895, p. 139.

[h] Buletinul camerei de comerciu si industrie din Bucuresci, 1900.

Thomson and Ballantyne,[a] in order to give these results a comparative value, proposed to report them as specific temperature numbers. They showed that with acid varying from 95.4 to 99 per cent there was only a very slight difference in results when calculated to specific temperature numbers, the stronger acid giving slightly higher results. Sherman, Danziger, and Kohnstamm[b] showed that if there was a much greater variation in the strength of acid used there was also a wider variation in the specific temperature numbers. With acid varying in strength from 89 to 95 per cent they found an average of from 90 to 103 in specific temperature numbers on the same olive oil.

The Maumené numbers obtained by the writers on 32 samples of olive oil of known purity using different strengths of acid are given in Table IX.

TABLE IX.—*Maumené and specific temperature numbers of olive oils.*

Serial number.	Rise of temperature given with pure water.	Maumené number.	Specific temperature number.	Rise of temperature given with pure water.	Maumené number.	Specific temperature number.	Rise of temperature given with pure water.	Maumené number.	Specific temperature number.
	°C.			°C.			°C.		
962	33.3	31.4	94.3	....	....	....	43.9	43.5	99.1
960	33.3	29.8	89.4	....	....	....	43.9	44.7	101.8
934	33.3	29.6	88.9	....	....	....	43.9	42.5	96.8
836	34.0	30,0	88.2	37.7	36.6	97.0	45.6	48.2	105.7
931	34.0	29.8	87.6	....	....	....	45.6	44.4	97.4
932	34.0	30.6	90.0	37.7	33.2	88.0	45.6	44.4	97.4
933	34.4	27.4	79.6	37.7	33.4	88.6	45.6	43.5	95.4
935	34.4	27.1	78.8	....	....	....	45.6	44.2	97.0
955	34.4	27.5	79.9	....	....	....	45.6	44.2	97.0
961	34.4	28.7	83.4	....	....	....	45.6	43.5	95.4
936	34.4	28.6	83.1	....	....	....	45.6	44.8	98.2
959	....	....	....	35.6	28.8	80.9	47.5	46.2	97.2
953	....	....	....	35.6	31.6	88.8	47.5	47.0	99.0
954	....	....	....	35.6	31.8	88.0	47.5	48.3	101.7
956	....	....	....	35.6	31.5	88.5	47.5	49.1	103.4
957	....	....	....	35.6	29.0	81.4	47.5	45.0	94.7
952	....	....	....	35.6	28.0	78.6	47.5	45.0	94.7
958	....	....	....	35.6	29.4	82.6	47.5	48.0	101.0
835	....	....	....	37.7	35.9	95.2	45.5	44.8	98.5
838	....	....	....	37.7	37.7	100.0	45.5	49.5	108.8
837	....	....	....	37.7	35.4	93.9	45.5	45.0	98.9
840	....	....	....	37.7	37.5	99.5	45.5	48.2	105.9
839	....	....	....	37.7	38.9	103.1	45.5	48.4	106.4
834	....	....	....	38.5	36.1	93.8	47.5	46.2	97.2
833	....	....	....	38.5	36.8	96.5	47.5	48.0	101.0
832	....	....	....	38.5	37.1	96.4	47.5	47.6	100.1
831	....	....	....	38.5	36.0	93.5	47.5	46.6	98.1
795	....	....	....	38.5	37.7	97.8	47.5	50.0	106.2
796	....	....	....	38.5	39.1	101.5	47.5	52.1	109.7
797	....	....	....	38.5	38.4	99.7	47.5	50.2	105.6
798	....	....	....	38.5	39.1	101.5	47.5	51.0	107.3
Maximum	34.4	31.4	94.3	38.5	39.1	103.1	47.5	52.1	109.7
Minimum	33.3	27.1	78.8	35.6	28.0	78.6	43.9	42.5	94.7
Difference	1.1	4.3	15.5	2.9	11.1	24.5	3.6	9.6	15.0

The results given in this table show that when there is a wide difference in the strength of acids used the specific temperature numbers are not of much value for comparative purposes, but if there is only a slight difference in strength they give very satisfactory results. The variation is least when a strong acid is used, giving a rise of temperature with water of 43.9° C. to 47.5° C., showing that the Maumené number obtained on olive oils with strong acid is more uniform than with the weaker acid. However, the strong acid is not satisfactory for use on seed oils, as the reaction becomes too violent and the oil is broken up. With seed oils a weaker acid was used, as suggested by Sherman, Danziger, and Kohnstamm.[a]

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[a] Jour. Soc. Chem. Ind., 1891, **10**: 233.

[b] Jour. Amer. Chem. Soc., 1902, **24**: 266.

The following table gives the results obtained in this laboratory on various oils. The strongest acid was used on the olive and lard oils, and a weaker acid on the seed oils.

TABLE X.—*Maumené and specific temperature numbers of various oils.*

Kind of oil.	Maumené number.	Specific temperature number.[a]	Kind of oil.	Maumené number.	Specific temperature number.[a]
California olive	42.5 to 52.0	94.7 to 109.7	Almond	45.3	117.6
Italian olive	42.5 to 49.1	95.6 to 104.7	Sesame	61.3	170.8
Lard	46.5 to 47.8	103.3 to 106.2	Sunflower	60.0	166.7
Peanut	61.0 to 63.2	135.5 to 140.2	Maize	75.2 to 89.2	190.2 to 212.5
Mustard	61.0 to 79.4	130.9 to 190.3	Cotton seed	66.2 to 73.4	172.4 to 191.1
Rape	54.5 to 67.8	135.0 to 152.5	Poppy	75.8 to 85.5	213.0 to 237.0

[a] Acids of quite widely differing strengths were used in these determinations.

IODIN ABSORPTION.

The method used for this determination was the modified Hübl method, as adopted by the Association of Official Agricultural Chemists,[b] in which the iodin solution is allowed to stand in contact with the oil for three hours.

The iodin number was found to be exceedingly variable. On 42 samples of California oil examined the range was 78.5 to 89.8, with an average of 85.1. Blasdale[c] found a range of from 80 to 86.5, with an average of 84. Colby[d] gives a range of 77.7 to 93.5 for California oils of known purity. On 17 samples of Italian oils the variation was from 79.2 to 86.1, with an average of 81.5. De Negri and Fabris[e] found on 88 samples of pure Italian oils a range of from 79.1 to 89.8, with an average of 83.7. Milliau[f] found for French oils a range of from 84 to 85, and for the oils of southern Europe and Tunis a range of from 81.4 to 85.2. Later[g] he found on Tunis oils a range of 79.3 to 89.5, with an average of 85, figures which are very similar to the results obtained on California oils. From these results it would seem that the range of iodin numbers on California oils is from 78 to 90, a little wider than that found on French oils, but about the same as on Italian and Algerian oils.

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[a] Jour. Amer. Chem. Soc., 1902, **24**: 266.

[b] U. S. Dept. Agr., Division of Chemistry Bul. 46, p. 50; Bul. 65, p. 24.

[c] Jour. Amer. Chem. Soc., 1895, **17**: 935.

[d] California Agr. Expt. Sta. Rept., 1897–98, p. 168.

[e] Annali del Labor. chim. centr. delle Gabelle, vol. 1, Gli Olii, pt. 1, p. 38.

[f] Bul. du Ministère de l'Agriculture, 1895, p. 139.

[g] Milliau, Bertainchand, and Malet, Rapport sur les huiles d'olive de Tunisie, 1900.

Table XI gives the range of iodin values found in this laboratory and by other analysts on various oils of known purity. With all these oils this range is extremely wide, and for this reason the determination of the iodin value is by itself of little value as a means of detecting adulteration. Only in cases of gross adulteration could it be conclusive. By a careful selection of the oils to be used in making the mixture, large proportions of either lard or peanut oil could be used without producing an abnormal iodin value. As is shown in Table XVII, page 26, the iodin value increases as the solid fatty acids and the melting point of the fatty acids decrease. It is only when this factor is determined, in connection with these other factors and with the iodin value of the liquid fatty acids, that it may serve as a valuable indication of the purity of an oil.

TABLE XI.—*The Hübl number of various oils.*

Kind of oil.	Results obtained in the Bureau of Chemistry.	Compiled results.	Kind of oil.	Results obtained in the Bureau of Chemistry.	Compiled results.
California olive	78.5 to 89.8	77.7 to 93.5	Cotton-seed	103.8 to 110.9	106.5 to 110.7
Italian olive	79.2 to 86.1	79.0 to 89.8	Sesame	106.6	102.0 to 112.0
French olive		84.1 to 84.5	Rape	92.5 to 101.2	94.1 to 106.2
Algerian olive		79.3 to 89.5	Mustard	98.4 to 113.0	92.1 to 106.5
Lard	69.7 to 77.2	60.0 to 82.0	Maize	116.7 to 123.3	111.1 to 123.9
Almond	96.2	93.0 to 102.0	Sunflower	104.1 to 108.3	119.0 to 135.0
Peanut	87.8 to 96.3	85.6 to 105.0	Poppy	133.2 to 134.9	130.5 to 141.0

The Hübl method has never been considered entirely satisfactory, owing to the poor keeping quality and the slowness of reaction of the iodin solution used. Recently two new solutions have been proposed as substitutes, both of which, it is claimed, remain practically unchanged for several months[a] and are more rapid in their reaction than the Hübl solution. The Wijs[b] solution consists of iodin mono-chlorid dissolved in glacial acetic acid; the other, proposed later by Hanus,[c] is a solution of iodin mono-bromid in glacial acetic acid. Comparative tests of these two solutions with the Hübl solution, made upon a large number of olive and other oils, verify the claims made for these new solutions both as regards their keeping qualities and their rapidity of action. A comparison of these methods is given in Table XII.

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[a] Tolman and Munson, Jour. Amer. Chem. Soc., 1903, **25**: 244.

[b] Ber. 1898, **31**: 752.

[c] Ztschr. Nahr. u. Genussm., 1901, **4**: 913.

TABLE XII.—*Iodin numbers of olive oils.*

Laboratory No.	Hübl number (3 hours).	Wijs number (30 minutes).	Hanus number (30 minutes).	Difference between Wijs and Hübl numbers.	Difference between Hanus and Hübl numbers.
795	89.7	90.9	90.4	+1.2	+0.7
796	89.7	90.6	90.0	+ .9	+ .3
797	89.8	91.4	90.0	+1.6	+ .2
798	89.7	91.1	90.4	+1.4	+ .7
960	80.9	82.5	81.7	+1.6	+ .8
833	84.8	86.7	86.5	+1.9	+1.7
831	85.2	86.7	85.9	+1.5	+ .7
832	84.9	86.5	85.9	+1.6	+1.0
834	84.5	86.1	85.1	+1.6	+ .6
936	81.8	82.8	82.8	+1.0	+1.0
933	80.6	81.6	81.1	+1.0	+ .5
835	82.7	83.1	82.6	+ .4	− .1
962	81.2	81.5	80.9	+ .3	− .3
955	82.6	83.6	83.1	+1.0	+ .5
952	81.3	82.2	81.8	+ .9	+ .5
840	86.3	87.8	86.7	+1.5	+ .4
935	80.5	80.9	81.6	+ .4	+1.1
953	79.2	79.9	80.6	+ .7	+1.4
958	81.8	83.1	81.8	+1.3	0.0
959	81.4	83.4	81.9	+2.0	+ .5
954	86.1	87.7	86.3	+1.6	+ .2
934	81.1	82.4	82.3	+1.3	+1.1
956	84.5	85.9	85.6	+1.4	+1.1
932	80.5	82.0	81.4	+1.5	+ .9
961	80.8	82.3	81.1	+1.5	+ .3
836	86.0	87.7	86.6	+1.7	+ .6
931	80.7	82.2	81.5	+1.5	+ .8
838	86.1	87.9	87.1	+1.8	+1.0
839	89.0	91.3	89.9	+2.3	+ .9
837	84.0	....	84.9	....	+ .9
673	83.3	84.1	82.6	+ .8	− .7
841	86.9	88.0	87.3	+1.1	+ .4
842	87.2	88.1	87.1	+ .9	+ .1
843	85.1	86.6	85.6	+1.5	+ .5
844	84.2	85.0	84.2	+ .8	0.0
1091	81.9	83.4	82.0	+1.5	+ .1
Average (36 samples)	....	....	....	+1.3	+0.60
Maximum	....	....	....	2.3	1.7
Minimum	....	....	....	0.3	0.0

Table XII gives the results obtained upon olive oils and Table XIII the results obtained upon other nondrying oils, semidrying, and drying oils. The values obtained with olive oils are slightly higher when the Wijs or the Hanus solution is used, but the difference is not sufficient to materially change the standard already existing for this oil. With other oils having a low iodin value closely agreeing results were obtained by the three methods, as shown in the values obtained for oleo oil, butter, and lard. With mustard and rape oil of the nondrying oils, and linseed oil of the drying oils, much higher results were obtained by the Wijs and the Hanus methods, and the use of either of these will necessitate the establishment of new standards.

TABLE XIII.—*Iodin numbers of various oils and fats.*

Laboratory No.	Oils and fats.	Hübl number (3 hours).	Wijs number (30 minutes).	Hanus number (30 minutes).	Difference between Wijs and Hübl numbers.	Difference between Hanus and Hübl numbers.
	FATS.					
	Cocoanut	8.93	9.05	8.60	+ 0.12	− 0.33
	Konut[a]	6.09	6.43	6.40	+ .34	+ .31
1170	Butter	35.3	36.2	35.3	+ .90	+ .00
1168	do	34.8	35.9	35.4	+ 1.10	+ .60
	Oleo	42.6	43.5	43.3	+ .90	+ .70
	Oleomargarine	53.6	53.5	52.3	− .10	− 1.30
	do	52.8	53.7	52.2	+ .90	− .60
	do	52.5	52.9	52.0	+ .40	− .50
	do	66.3	66.0	64.8	− .30	− 1.50
	NONDRYING OILS.					
23606	Lard	69.3	70.5	69.8	+ 1.20	+ .50
487	do.[a]	73.7	74.5	73.9	+ .70	+ .20
1181	Magnolia	81.7	79.4	78.9	− 2.30	− 2.80
1182	do	76.1	75.6	74.0	− .50	− 2.10
772	Peanut	96.3	99.0	97.4	+ 3.00	+ 1.10
1149	do	94.5	95.2	94.1	+ .70	− .10
492	do.[b]	107.7	109.5	107.7	+ 1.80	+ .00
770	Mustard	110.4	118.5	115.5	+ 8.10	+ 5.10
771	do	113.0	118.2	116.8	+ 5.20	+ 3.80
776	do	98.4	104.3	103.8	+ 5.90	+ 5.40
486	do.[a]	103.5	112.5	110.2	+ 9.00	+ 6.70
495	do.[a]	106.4	117.3	114.8	+10.90	+ 8.40
775	Rape	101.3	105.7	105.2	+ 4.40	+ 3.80
490	do.[a]	100.2	104.1	102.8	+ 3.90	+ 2.60
	SEMIDRYING AND DRYING OILS.					
	Sunflower	106.4	109.2	107.7	+ 2.80	+ .80
1159	Cottonseed	103.8	105.3	105.2	+ 1.50	+ 1.40
1160	do	106.2	107.3	107.8	+ 1.10	+ 1.60
1161	do	104.8	106.2	106.7	+ 1.40	+ 1.90
489	Sesame[a]	106.4	107.0	106.5	+ .60	+ .10
444	Maize[a]	119.0	123.2	120.2	+ 4.20	+ 1.20
491	do.[a]	119.0	122.2	119.6	+ 3.00	+ .40
777	do	123.3	129.2	126.0	+ 5.80	+ 2.70
493	Poppy[a]	133.4	135.2	132.9	+ 1.80	− .50
774	do	134.9	139.1	138.4	+ 4.20	+ 3.50
1162	Linseed[a]	169.8	186.5	184.5	+16.70	+14.70
1188	do.[c]	179.5	188.7	183.7	+ 9.20	+ 4.20

[a] Commercial oils.
[b] Adulterated with cotton-seed oil.
[c] Four hours for Hübl determination.

As will be seen from Table XIV, the length of time involved in the determination is greatly lessened by the use of the Wijs and the Hanus solutions. In case of olive oils the reaction is complete in fifteen minutes; cotton seed, sesame, mustard, and other nondrying and semidrying oils require thirty minutes; while rape and linseed oils require one hour for complete absorption.

TABLE XIV.—*Time necessary for absorption of iodin.*

Laboratory No.	Kind of oil or fat.	Iodin numbers by Hanus method.			Iodin numbers by Wijs method.		
		15 minutes.	30 minutes.	1 hour.	15 minutes.	30 minutes.	1 hour.
773	Cocoanut	8.7	8.6	....	....	....	....
22077	Konut	6.4	6.4	....	....	....	....
1170	Butter	35.2	35.4	....	....	....	....
1168	.....do	35.4	35.5	....	35.8	35.9	36.0
	Oleo	43.2	43.4	....	....	....	....
	Oleomargarine	52.4	51.8	....	....	....	....
	.....do	51.9	52.1	....	52.9	....	53.0
	.....do	64.6	65.1	....	....	....	....
	.....do	52.2	52.4	....	....	....	....
	.....do	80.4	80.5	....	....	....	....
23606	Lard	70.0	69.7	....	....	....	....
798	Olive	90.1	90.4	90.3	90.0	91.2	90.9
960	.....do	82.1	81.7	82.2	82.6	82.6	82.5
833	.....do	86.5	86.5	86.5	86.5	86.7	87.2
772	Peanut	96.0	97.4	97.7	98.8	99.0	99.0
777	Mustard	125.0	126.0	126.0	126.5	126.5	128.0
771	.....do	....	119.4	120.0	....	118.0	118.2
776	.....do	....	105.0	105.0	....	104.3	104.6
775	Rape	....	107.4	107.5	....	105.8	105.7
770	Maize	....	116.8	117.6	117.1	116.1	118.5
774	Poppy	136.8	137.4	138.4	138.8	138.9	139.1
1162	Linseed	....	186.3	186.2	183.8	190.2	188.7

These results show but little choice between the two new solutions, the Hanus solution giving results slightly closer to the figures obtained by the Hübl method.

The Wijs solution is prepared by dissolving 13 grams of pure iodin in a liter of glacial acetic acid which does not reduce a solution of bichromate of potash in sulphuric acid; a current of pure chlorin gas is run into the solution until the halogen content is doubled. A slight excess of iodin is recommended. The change of color in the solution as the end point is approached is quite marked.

The Hanus solution used is made by dissolving 13 grams of iodin in a liter of glacial acetic acid and then adding sufficient bromin to double the halogen content; about 3 cc are sufficient.

The method of using these solutions is the same as with the Hübl solution except that an excess of unabsorbed iodin of at least 70 per cent of the total titer is necessary; and only 10 cc of the 20 per cent potassium iodid solution are used, the solution being thoroughly mixed before the water is added. In the above work the absorption was allowed to continue thirty minutes, as some of the adulterants of olive oil require a slightly longer time than the oil itself to complete the absorption.

Great care must be taken that no change of temperature occurs while measuring the solutions. The high coefficient of expansion of acetic acid (0.00115 for 1° C.) will cause an appreciable error if even a slight change of temperature takes place. If 40 cc of these solutions are measured, a change in temperature of ± 1° C. will give a change of ∓ 0.11 cc in the amount of N/10 thiosulphate used to neutralize them. Table XV shows how great the variation in titer may be in a change of temperature of 11° C.

TABLE XV.—*Influence of temperature on titer.*

Temperature.	40 cc iodin-mono-bromid in N/10 thio-sul-
°C.	cc.
16	92.05
17.5	91.85
18.5	91.80
21.5	91.35
24.5	91.10
27	90.80
+11	—1.25

The results of this investigation show that either of the two new methods gives satisfactory results and that both solutions are decided improvements over the Hübl solution, not only in possessing much better keeping qualities, but also in rapidity of action. The Hanus solution gives results somewhat closer to those obtained by the Hübl method, and is much more easily prepared than the Wijs solution.

SAPONIFICATION OR KOETTSTORFER NUMBER.

The saponification number, or number of milligrams of potassium hydroxid needed to saponify 1 gram of fat, was determined as directed by the Association of Official Agricultural Chemists.[a] The determination has little value in detecting adulteration of olive oil with any of the animal or vegetable oils except rape or mustard. Its chief value is in the detection of mineral oils.

Table XVI, giving the limits on various oils, shows the uniformity of the Koettstorfer number of the ordinary salad oils, except rape and mustard oil. The variation on the oils other than rape and mustard is from 185.0 to 197.7, while the variation on olive oils is from 185.0 to 194.6.

TABLE XVI.—*Saponification or Koettstorfer number of various oils.*

Kind of oil.	Results obtained in Bureau of Chemistry.	Compiled results.	Kind of oil.	Results obtained in Bureau of Chemistry.	Compiled results.
California olive ..	189.3 to 194.6	187.0 to 193.5	Peanut...........	188.8 to 196.0	189.3 to 196.0
Italian olive......	189.7 to 192.0	185.0 to 192.3	Cotton seed	196.0 to 198.5	191.0 to 196.6
Lard..............	195.3 to 197.7	189.5 to 196.0	Sesame...........	190.7	188.4 to 194.6
Rape	174.1 to 176.6	167.7 to 183.0	Maize	189.9 to 193.4	188.0 to 193.4
Mustard	173.0 to 182.8	170.2 to 174.7	Sunflower........	191.2 to 192.3	188.0 to 197.6
Almond..........	192.5	183.0 to 191.7	Poppy seed	190.2 to 193.8	192.8 to 194.0

MELTING POINT OF FATTY ACIDS.

The method used for this determination was that described in the Provisional Methods for Food Analysis.[b] A variation was found on

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[a] U. S. Dept. Agr., Division of Chemistry Bul. 46, p. 48.

[b] U. S. Dept. Agr., Bureau of Chemistry Bul. 65, p. 31.

California oils of known purity of from 19.2° to 31° with an average of 22.5°. Blasdale[a] found the variation on eleven samples of California oils to be from 21° to 26°, and Colby[b] gives a range of from 21° to 28°. On Italian oils the writers found a variation of from 21° to 29.3°. Other published results give a variation of 23.7° to 29°. Milliau[c] reports on French oils 24° to 26°, but on Algerian[d] oils gives a range of from 24.8° to 38°. This shows that Algerian oils are quite different from the European and Californian products.

Table XVII, prepared from Table XXXV, shows the relation between the Hübl number, percentage of solid fatty acids, and melting point of fatty acids in California olive oils. The writers found that in a general way the same relation held good for Italian oils. Milliau[d] did not find this relation to hold on oils from Tunis. He found oils with a Hübl number of 88 and a melting point of fatty acids of 37° C., and concluded from this that there were fatty acids present more unsaturated than oleic acid.

TABLE XVII.—*Relation between Hübl number, solid fatty acids, and melting point of fatty acids (California oils).*

Serial No.	Hübl number.	Solid fatty acids.	Melting point of fatty acids.	Serial No.	Hübl number.	Solid fatty acids.	Melting point of fatty acids.
		Per cent.	°*C.*			*Per cent.*	°*C.*
23462	79.9	10.91	31.0	23463	85.6	4.92	21.3
23461	83.0	7.62	28.0	23460	85.7	6.27	23.4
23459	82.9	5.70	25.0	23457	86.2	3.39	21.1
23456	84.3	7.23	23.4	23458	88.2	4.42	23.5
23692	85.6	5.12	22.6	23605	88.5	2.43	20.2

The melting points of the fatty acids of peanut oil, cotton-seed oil, and lard oil are so much higher than those of the mixed acids of olive oil that any considerable addition of any one of these oils to olive oil would produce an abnormal melting point. This is especially true of lard oil and cotton-seed oil. Sesame oil varies but little from olive oil in respect to the melting point of its fatty acids, but oils of rape seed, mustard seed, sunflower, poppy seed, and maize are appreciably lower. Since, however, none of these would probably be mixed with any but olive oil or some one of the oils having a higher melting point than olive oil, this difference would be of little value in the detection of their substitution for olive oil. The determination of the melting point in itself has little value in the detection of adulteration, but is of decided value when taken in connection with other factors in judging whether or not the oil is normal. The melting points of the fatty acids of various oils as determined in this laboratory and as found in the literature on the subject are given in Table XVIII.

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[a] Jour. Amer. Chem. Soc., 1895, 17; 935.

[b] California Agr. Expt. Sta. Rept., 1897-98, p. 169.

[c] Bul. du Ministère de l'Agriculture, 1895, p. 139.

[d] Milliau, Bertainchand, and Malet, Rapport sur les huiles d'olive de Tunisie, 1900.

TABLE XVIII.—*Melting point of fatty acids of various oils.*

Kind of oil.	Results obtained in Bureau of Chemistry.	Compiled results.	Kind of oil.	Results obtained in Bureau of Chemistry.	Compiled results.
	°C.	°C.		°C.	°C.
California olive	19.2 to 31.0	21.0 to 28.0	Sunflower	21.0	17.0 to 24.0
Italian olive	21.6 to 29.3	23.7 to 29.0	Maize	21.6 to 23.0	16.0 to 22.4
French olive	....	24.0 to 26.0	Poppy	25.4 to 25.8	20.0 to 21.0
Algerian olive	....	24.8 to 38.0	Sesame	27.4	21.0 to 40.0
Almond	23.2	12.0 to 14.0	Peanut	33.2 to 37.6	26.0 to 36.4
Mustard	20.8 to 21.5	15.0 to 21.5	Lard	33.2 to 38.4	33.0 to 38.4
Rape	20.0 to 21.9	15.5 to 22.0	Cotton seed	35.5 to 39.6	32.0 to 43.0

SOLID FATTY ACIDS.

By solid fatty acids are meant those whose lead salts are insoluble in ether. The method of determination used was that described in the Provisional Methods for Food Analysis.[a] From these results, calculating the average total fatty acids present as 95.5 per cent,[b] the percentage of liquid fatty acids can be obtained. As the latter are the unsaturated fatty acids and alone absorb the iodin, the percentage of these acids and their iodin value gives a fair idea of the nature of the oil under consideration. Some of the oils of Tunis must contain fatty acids more unsaturated than oleic to give the high iodin number, and also the high per cent of solid fatty acids found by Milliau, Bertainchand, and Malet.[b]

The iodin number of the oil multiplied by 100 and divided by the per cent of liquid fatty acids will give their iodin number, providing the solid fatty acids are saturated. The calculated results agree fairly well with those obtained by experiment. These results show that in olive oils the unsaturated fatty acids are variable in their composition and can not be considered as pure oleic acid.

From Table XXXIII it is seen that in lard oil the unsaturated fatty acids have practically the same iodin number as the unsaturated acids of olive oil. Rape-seed oil does not differ much in this respect from olive oil. Mixtures of lard and cotton-seed oils which would give a normal iodin number for olive oil would have a much higher iodin number for the liquid acids. The relations between the solid fatty acids, the iodin number of the oil, and the liquid fatty acids, are of great value in identifying an oil of unknown origin. These factors for various oils are given in Table XIX. It can be seen from this table that the mixing of these oils with olive oil will change the figures on the mixture in a number of ways. An addition of maize oil would increase the iodin number of both the oil and the liquid fatty acids, but would not affect the solid fatty acids. Cotton-seed oil would increase all of these figures.

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[a] U. S. Dept. Agr., Bureau of Chemistry Bul. 65, p. 28.

[a] Rapport sur les huiles d'olive de Tunisie, 1900.

TABLE XIX.—*The Iodin numbers and the per cent of solid fatty acids of various oils.*

Kind of oil.	Hübl number.	Iodin number of liquid fatty acids.	Solid fatty acids.	Kind of oil.	Hübl number.	Iodin number of liquid fatty acids.	Solid fatty acids.
			Per cent.				*Per cent.*
Poppy	134.9	151.7	6.67	Sunflower	108.3	117.8	3.67
Maize	123.3	139.8	7.44	Do	104.1	113.8	4.12
Do	119.7	134.8	6.70	Rape	92.5	96.9	.12
Do	119.2	134.5	6.98	Do	101.2	107.5	1.43
Mustard	113.0	121.1	2.32	Sesame	97.9	115.4	10.70
Do	110.4	116.8	1.06	Lard	75.9	98.9	18.90
Do	98.4	103.0	Trace.	Do	69.7	101.3	26.68
Cotton-seed	108.3	141.5	19.04	Do	72.5	97.9	21.43
Do	105.8	136.3	17.87	Do	77.2	101.8	19.30

The addition of mustard oil would increase the iodin numbers, but decrease the solid fatty acids. Lard oil would decrease the Hübl number and increase the solid fatty acids, but would not affect the iodin number of the liquid fatty acids. In many cases, however, especially if but small amounts were added to olive oil, the changes in the factors would be so slight as to give no indication of any adulteration.

FREE FATTY ACIDS.

The free fatty acids were determined by the method described in the Provisional Methods for Food Analysis[a] and the results calculated as oleic acid. One cubic centimeter of deci-normal alkali is equal to 0.0282 gram of oleic acid. This determination is of considerable value, in that it gives some idea of the condition of the oil. As a rule this figure increases with age and may be to some extent an index of the rancidity of the oil. The amount of free acid greatly affects the physical constants of oils as has already been shown. All oils have some free acid present, even when freshly pressed. Its amount probably depends on the condition of the fruit or seed and the processes of manufacture. Milliau, Bertainchand, and Malet[b] found as high as 3.9 per cent of free acid in freshly pressed olive oil, probably due to the overripe or decayed condition of the olives.

With a number of cold-pressed seed oils manufactured in this laboratory the determination of free fatty acids was made within three days after the oils were pressed, with the following results: Sunflower oils, 0.18 and 1.72 per cent; peanut oil, 0.04 per cent; mustard-seed oil, 1.13 per cent; and rape-seed oil, 0.63 per cent. These figures show the presence of considerable amounts of free acids in the seeds. In Table XX are given the data showing the quantity of free acid in a number of samples of olive oil of known history. Nos. 22617, 22714, and 23126 all show an acidity that might be considered normal for fresh oils, although eight and ten years old. On the other hand Nos. 22618 and 22619, which were three years and one year old, respectively, show an excessive amount of free acid. It therefore follows that the time factor has but little influence upon the acidity, while No. 22619 indicates that high temperature favors the formation of the free acids.

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[a] U. S. Dept. Agr., Bureau of Chemistry Bul. 65, p. 27.

[b] Rapport sur les huiles d'olive de Tunisie, 1900.

TABLE XX.—*Free acids in olive oils.*

Serial No.	Free fatty acids as oleic.	History of sample.
	Per cent.	
22617	0.36	Made in 1893 from Redding Picholine olives.
22618	8.21	Made in 1898 from mixed varieties of olives.
22619	44.40	Made in 1900 from Manzanillo olives by strongly heating the fruit.
22714	1.26	Second pressing, 10 years old.
22715	2.73	Poor quality of oil, not an edible oil.
22126	1.40	South American oils exhibited at Chicago in 1893.
22127	4.33	

Eleven samples of pure California olive oils, all of which were considered fresh oils, gave an average content of free acid of 1.16 per cent, and the extremes were 0.59 per cent and 2.24 per cent. Twelve samples of commercial California olive oils in which no added oil was detected contained an average of 2.40 per cent free acids, and this figure is more nearly in accord with the averages of the French and Italian commercial oils than is the average given for the California oils of known purity. This is undoubtedly due to the fact that previous to leaving the storeroom of the manufacturer the oil is kept under more favorable conditions of light and temperature than it receives after entering the retail stores.

By reference to Table XXXIII it is seen that lard oil, peanut oil, and the various seed oils show an appreciable content of free acids, differing in this respect but little from olive oils. Cotton-seed oil alone is an exception. This oil is refined by use of alkali, and so shows but small amounts of acidity and yields but little upon long standing.

The rancidity of oils and fats has been the subject of extensive study by various investigators, and opinions regarding its causes are extremely varied. Lewkowitsch[a] states that pure, nondrying oils when protected from air and light remain unchanged at ordinary temperatures; on exposure to light and air they undergo change, consisting of the development of butyric, isobutylacetic, and other volatile acids in small quantities, an increase in free acids, and the acquirement of a disagreeable smell and acrid taste. These changes the author considers due to direct oxidation by the oxygen of the air, this action being intensified by sunlight.

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[a] Chem. anal. oils, fats, and waxes, 2d ed., p. 10.

Ballantyne[a] has shown that in many instances rancidity exists long before the formation of free acids, and that on the other hand free acids may be liberated long before the oil becomes rancid. A. Schmidt[b] states that an oil may have a high acidity without being rancid, and that the oil becomes rancid upon partial or complete oxidation of the free glycerin to aldehydes and ketones. Scalé[c] found that olive oil and lard contained no water soluble volatile acids in a fresh condition, but these acids developed with age and consequent rancidity of the oil and fat. Browne[d] has made a study of the changes incident to the development of rancidity in butter fat and records some interesting results. Table XXI, taken from his work, shows the effect of the development of rancidity upon the various values of the oil. As the rancidity develops there is a decided increase in the acid, saponification, ether, and Reichert values, a marked decrease in the iodin value and the percentage of insoluble acids, and a slight decrease in the percentage of glycerin.

TABLE XXI.—*Effect of development of rancidity upon butter fat* (*Browne*).

No. of sample.	Condition.	Acid value.	Saponification number.	Ether number.	Reichert number.	Hübl number.	Oleic acid (calculated).	Insoluble acids.	Glycerol.
							Per cent.	*Per cent.*	*Per cent.*
0	Fresh	0.48	228.1	227.6	15.63	34.95	38.79	....	....
0	1 week old	1.28	230.3	229.0	15.80	34.55	38.35	....	....
0	1 month old	10.90	241.0	230.1	17.00	28.40	31.52	....	....
0	2 months old	28.84	260.0	231.2	18.75	14.35	15.93	....	....
0	4 months old	30.00	262.1	232.1	19.80	11.15	12.38	....	....
0	8 months old	35.38	269.3	233.9	21.13	8.56	9.49	....	....
1a	Fresh	.45	229.9	229.5	....	33.93	37.66	87.20	12.54
1b	Rancid	1.22	232.3	231.1	....	29.96	33.26	86.80	12.40
2a	Fresh	.50	223.9	223.4	....	34.49	38.28	88.96	12.21
2b	Rancid, 1 month old	7.09	233.7	226.6	....	28.69	31.85	85.06	12.02
3a	Fresh	.55	232.6	232.1	....	29.56	32.81	86.41	12.69
3b	Rancid, 2 months old	11.73	247.7	236.0	....	19.76	21.93	80.42	12.35
4a	Fresh	.51	225.6	225.1	....	34.92	38.76	88.46	12.33
4b	Rancid, 3 months old	14.80	245.3	230.5	....	22.55	25.03	81.15	11.67

Rancidity may affect the physical and chemical values of oils and fats in several ways. If large amounts of free acid are formed and little oxidation takes place the specific gravity and index of refraction may be lowered and the iodin number very little affected. If oxidation has taken place there will be an increase in the specific gravity, index of refraction, and saponification number, and a decrease in the iodin number.

From the foregoing it is apparent that oils are extremely susceptible to change under the influence of light, air, and heat, and that deterioration is in a large measure prevented by the exclusion of air and sunlight and by proper temperature conditions. These facts are of material importance, as the pleasant flavor and agreeable odor of pure, fresh olive oil may be easily destroyed upon the development of rancidity, while if kept under proper conditions the oils will remain practically unchanged for a long time. From the chemist's standpoint rancidity may so affect the values of an oil as to make it abnormal in many respects, and in the interpretation of analytical data this fact must be given due consideration.

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[a] Jour. Soc. Chem. Ind., 1891, **10:** 29.

[b] Ztschr. Anal. Chem., 1898, **37:** 301.

[c] Ztschr. Nahr.-Unt. Hyg. u. Waarenk., 1896, **10:** 239.

[d] Jour. Amer. Chem. Soc., 1899, **21:** 975.

OLIVE OIL SUBSTITUTES.

Cotton-Seed Oil.

Cotton-seed oil is the chief adulterant of and substitute for olive oil in this country. It is used to a large extent under the name of salad oil, but is also frequently sold as olive oil, with which it is extensively mixed. The well-refined oil has a pleasant though characteristic taste and odor, and is much less inclined to become rancid than are olive oil and other oils used for salad purposes. The cotton-seed oil sold for salads is refined in part by the use of alkali, and this treatment removes all free, fatty acids. The samples of cotton-seed oil found upon the market all showed very low percentages of free, fatty acids due to the above treatment and to the tendency of this oil to remain stable. One sample of unpurified, cold pressed cotton-seed oil examined had 2.17 per cent of free acids, but practically all of this amount existed in the seed at the time of pressing, as free acids were determined within a few days after the oil was pressed.

Table XXII.—*Cotton-seed oil.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
De Negri and Fabris:[a]		*Degrees.*						° *C.*	° *C.*	*Per ct.*
Minimum	0.9230	....	....	50.0	....	106.5	191.8	34.0	....	......
Maximum	.9250	....	....	53.0	....	110.7	194.7	39.0	....	......
Tolman and Munson:[b]										
Minimum	.9226	72.3	1.4737	66.2	172.4	103.8	....	....	....	0.04
Maximum	.9236	75.6	1.4757	73.4	191.1	110.9	....	....	....	2.17
Allen:[c]										
Minimum	.9160	....	....	74.0	163.0	102.0	191.0	35.0	32.2	......
Maximum	.9800	....	....	77.0	170.0	111.0	196.6	40.0	37.6	......
Lewkowitsch:[d]										
Minimum	.9250	....	1.4747	75.5	....	106.0	192.5	35.0	30.5	......
Maximum						108.0		43.0	35.2	......
Twenty-two other analysts:[e]										
Minimum	.9216	....	....	55.0	....	104.5	191.0	32.0	....	......
Maximum	.9362	....	....	71.0	....	114.0	195.0	43.0	....	......

[a] 10 samples. Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 92.
[b] 4 samples.
[c] Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 140.
[d] Oils, Fats, and Waxes, p. 374.
[e] Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 92.

Table XXII is a compilation of the results of a large number of analyses, and gives the limits which have been obtained for this oil. Some of these figures are so extraordinary that the oils from which they were obtained can hardly be considered otherwise than abnormal. In this bulletin the term "normal oils" is used to indicate those which are not so rancid as to be unfit to be eaten or which have not been oxidized by artificial means. On rancid or oxidized oils results can be obtained which are entirely different from those which would be found on fresh oils, and in this report salad oils only are considered. Cotton-seed oils with a specific gravity as low as 0.9160 and as high as 0.9360 are most likely abnormal. Such figures can hardly be considered of any value unless further data as to free acids and condition of the oils are known. In this connection samples Nos. 13235 and 23656 of peanut oil in Table XXXIII serve as examples. The first oil was what is called a "blown" oil, and the results obtained with it are entirely abnormal. It has a specific gravity of 0.9364, an iodin number of 77, and saponification number of 199. The second has a specific gravity of 0.9155, but has 13.51 per cent of free acid. Such an oil could not be used under any circumstances as salad oil, and certainly should not be considered in establishing limits for peanut oil.

When added in any considerable amounts to olive oil, cotton-seed oil is not difficult to detect, on account of its high specific gravity, iodin number, Maumené figure, and index of refraction. Moreover, there are the qualitative tests of Bechi, Halphen, and the nitric acid reaction to detect it in small amounts. The Halphen test[a] is extremely delicate, detecting 1 per cent or less of the unheated oil. This test is characteristic of cotton-seed oil, and if obtained in an olive oil is proof of its presence. But Holde and Pelgry[b] have shown that cotton-seed oil if heated for ten minutes to 250° C. loses the power of giving both the Halphen and Bechi tests. The writers found that it took twenty minutes to completely destroy the power to give the Halphen test, but the reaction was greatly weakened on oils heated a much shorter time. This heated oil if refined would lose the taste and odor due to heating and could be mixed with olive oil in small amounts without giving the Halphen, Bechi, or Milliau test. But the nitric acid reaction is apparently not affected in any way by this heating.

Tortelli and Ruggeri[c] have made an extensive study of heated cotton-seed oil in order to discover means of detecting it in olive oil. They found that it was necessary to heat the cotton-seed oil twenty minutes at 250° C. in order to completely destroy its power of reacting with Halphen or Bechi reagents. The values of the oil are not affected to any great extent by this heating, as is shown in Table XXIII, taken from their work.

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[a] U. S. Dept. Agr., Bureau of Chemistry Bul. 65, p. 32.

[b] Chem. Rev. Fett. u. Hartz. Ind., 1899, **6**: 67; Jour. Soc. Chem. Ind., 1899, **18**: 711.

[c] Annali del Lab. chim. cent. delle Gabelle, 1900, p. 249.

TABLE XXIII.—*Values of heated cotton-seed oil* (*Tortelli and Ruggeri*).

Temperature and time of heating.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Maumené number.	Hübl number.	Iodin number of liquid fatty acids.	Melting point of fatty acids.	Solidifying point of fatty acids.
		Degrees.				° *C.*	° *C.*
Original oil	0.9240	56.0	67.0	108.6	147.0	35.8	....
200° C. for 10 minutes	....	....	....	104.0	140.2	....	....
250° C. for 10 minutes	....	....	....	103.8	138.3	....	....
Original oil	.9240	56.0	67.0	109.9	147.7	35.8	32.2
200° C. for 10 minutes	.9230	56.5	67.0	108.3	142.7	35.9	32.4
250° C. for 10 minutes	.9244	56.5	66.0	107.5	141.5	36.0	32.5
250° C. for 20 minutes	.9243	57.5	65.0	106.9	140.7	36.2	32.5

The iodin number of the liquid fatty acids is affected by heating to a greater degree than any other value, being considerably lowered. Tortelli and Ruggeri propose the following method for the detection of the heated oil which depends on the fact that while such an oil will not give the Halphen or Bechi test, the reacting substance is not entirely destroyed, and by concentrating this a reduction of silver is obtainable.

The liquid fatty acids are separated as described under Muter's method.[a] The liquid fatty acids are treated in a test tube with a mixture of 1 cc of a 5 per cent aqueous solution of silver nitrate and 10 cc of 90 per cent alcohol, and heated to from 70° to 80° for several hours if necessary, in order to get a reduction of silver. By this method small quantities of heated cotton-seed oil in olive oil may be detected. Other reactions which will show the presence of heated cotton-seed oil are the nitric-acid test, using acid of specific gravity 1.375, and the Brullé[b] test, with nitric acid and egg albumen. The former was tried on some heated cotton-seed oil which would not react with the Halphen reagent, and a strong reaction was obtained with a mixture of 10 per cent of heated cotton-seed oil and 90 per cent of olive oil. Smaller amounts could easily be detected. The test is not as sensitive as the Halphen or Bechi test on untreated oils, but it is of great value for use with heated oils. It may also be of value in determining whether a weak test with the Halphen reagent is due to a small amount of unheated cotton-seed oil or a larger amount which has been heated enough to weaken the Halphen reaction. Some of the other oils react, however, with nitric acid. Sunflower oil, treated by this method, gives a deep brown color which can not be distinguished from the color obtained with cotton-seed oil. Peanut oil gives only a very slight test. Maize oil reacts very strongly, giving a peculiar reddish color that might be mistaken at first for the cotton-seed oil test.

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[a] U. S. Dept. Agr., Bureau of Chemistry Bul. 65, p. 28.

[b] R. Brullé, Comp. rend., 1893, **106**: 1017.

The Brullé[a] test for cotton-seed oil is also effective on oils heated to 250° C. It is conducted as follows: Place in a test tube 0.1 gram of dry albumen finely powdered and 2 cc of nitric acid (3 parts nitric acid, 1.40 specific gravity and 1 part water) and 10 cc of oil. Heat without stirring until the acid begins to boil, then cautiously agitate until the albumen is dissolved. Under this treatment olive oil will not show any change of color, while cotton-seed oil can be detected by the red coloration. Other oils, such as peanut, sunflower, and rape, are also colored by this process. Olive oil forms an elaidin after this treatment, which is very different from that given by the other oils, except lard oil, which, because of its content of oleic acid, acts very similarly.

PEANUT OIL.

Peanut oil is probably used as an adulterant of olive oil more extensively in Europe than in this country where cotton-seed oil is so common. The oil has a pleasant, nutty flavor, and makes a good salad oil, for which purpose it is quite extensively used in Europe under its own name. The values of peanut oil are so little different from olive oil that considerable quantities of it could be mixed with olive oil without being detected by these figures. Table XXIV gives the results compiled from published data on peanut oil.

TABLE XXIV.—*Peanut oil.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5°C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
Müntz, Durand, and Milliau:[a]		*Degrees.*						° C.	° C.	*Per ct.*
Minimum	0.9175									
Maximum	.9210	....	....	46.0	127.0	97.0	......	31.0	28.0	......
S. P. Sadtler:[b]										
Minimum	.9110	....	....	49.0	......	85.6	190.6	29.0	25.0	0.54
Maximum	.9200	....	....	56.7	......	95.0	194.0	34.0	32.0	.79
De Negri and Fabris:[c]										
Minimum	.9165	....	....	45.0	......	92.0	189.4	27.0	22.0	......
Maximum	.9200	....	....	51.0	......	100.0	193.0	31.0	25.0	......
Tolman and Munson:[d]										
Minimum	.9155	67.5	1.4707	46.5	129.1	87.8	188.8	33.2	......	.24
Maximum	.9188	71.3	1.4731	63.2	154.2	96.3	191.8	36.4	......	13.51
Twenty-one other analysts:[e]										
Minimum	.9160	....	....	44.0	......	85.6	189.3	26.0	23.0	......
Maximum	.9220	....	....	67.0	......	105.0	196.0	35.0	31.0	......
Maximum	.9220	....	....	67.0	154.2	105.0	196.0	36.4	32.0	13.51
Minimum	.9110	....	....	44.0	127.0	85.6	188.8	26.0	23.0	.24

[a] Bulletin du Ministère de l'Agriculture, 1895, p. 139.
[b] 4 samples. Amer. Druggist and Pharm. Rec., 1897, vol. 31, no. 5.
[c] 8 samples. Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 12.
[d] 5 samples.
[e] Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 12.

From this table it can be seen that oils could be selected for admixture which would not affect the values of olive oils to any degree except in case of very gross adulteration. Sample 499, Table XXXIII, a cold pressed oil prepared in this laboratory, is an example of this. It has a specific gravity of 0.9186, a Hübl number of 87.8 and 0.40 per cent of free acid. Such an oil could be mixed in almost any proportion with an olive oil of low iodin number and not give even a hint from the physical and chemical values of its presence. Practically the only test that is positive is the isolation of arachidic acid, which is present in peanut oil to the extent of from 3 to 5 per cent. In four samples of peanut oil examined in this laboratory from 3.41 to 4.24 per cent of the acid was found, having a melting point of from 72° to 73°. Allen[a] gives an average of 5 per cent. In Table XXXIII results are given on two samples of peanut oil, Nos. 13234 and 13235, which were examined to determine the effect of age. Both had been kept in stoppered bottles for eight years; No. 13235 was a heated oil. Both were exceedingly rancid to taste and smell, but No. 13234 had only 2.93 per cent of free acid, showing that acidity is not an index of rancidity. No. 13235 shows that great changes have been brought about in the oil in the process of heating.

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[a] Chem. Ztg. Rep. 1888, 12: 107.

Renard's[b] test is as satisfactory as any of the methods for detecting arachidic acid. Twenty grams of the oil to be tested should be taken so that enough of the arachidic acid can be separated to make the melting point which should be determined in every case, as some oils, such as cotton-seed and lard, will give a precipitate which resembles arachidic acid except that it has a lower melting point.

SESAME OIL.

Like peanut oil, sesame oil has a pleasant flavor and is well adapted to the adulteration of olive oil. However, it is not extensively used in this country, as the analyses of commercial samples show. Table XXV gives the results compiled from published data on sesame oil.

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[a] Com Org. Anal., 3d ed., vol. 2, pt. 1, p. 134.

[b] Comp. Rend. 1871, **73:** 1330. U. S. Dept. Agr., Bureau of Chemistry, Bul. 65, p. 33.

TABLE XXV.—*Sesame oil.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
De Negri and Fabris:[a]		*Degrees.*						° *C.*	° *C.*	*Per ct.*
Minimum	0.9230	......	......	63.0	......	106.8	188.4	23.0	20.0	......
Maximum	.9237	......	......	64.0	......	107.7	190.4	26.0	22.0	......
Tolman and Munson[b]	.9218	73.3	1.4742	61.3	170.3	97.9	190.7	27.4	......	0.44
Müntz, Durand, and Milliau[c]	.9230	......	......	54.0	150.0	104.0	......	26.0	22.0	......
Twenty-five other analysts:[d]										
Minimum	.9200	......	......	65.0	......	102.0	190.0	21.0	21.0	......
Maximum	.9250	......	......	68.5	......	112.0	194.6	40.0	34.0	......

[a] 6 samples. Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 82.
[b] 1 sample.
[c] Bulletin du Ministère de l'Agriculture, 1895, p. 139.
[d] Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 82.

The specific gravity, index of refraction, Maumené number, and iodin number of this oil are all materially higher than for olive oil, and serve as an indication of adulteration. The qualitative test of Baudouin and Villavecchia's modification of the same, are characteristic of sesame oil and permit of the certain detection of amounts as small as 2 per cent. It must always be remembered, however, that there are some olive oils from Italy and Tunis which give a reaction with this test which is difficult to differentiate unless comparative tests are carried on with sesame oil, when they can be readily distinguished. Milliau[a] states that when the fatty acids were used in making this test on Tunis oils the red coloration found with the oils themselves was not obtained.

MAIZE OIL.

Maize oil is produced in large amounts in the United States, and, owing to its cheapness, may find use as a substitute for olive oil. This product, which is a semidrying oil similar to cotton-seed oil, has thus far been used but little for salad purposes, only one sample being found upon the market. It possesses the flavor and odor characteristic of maize, and these qualities aid in its detection when not mixed with other oils. The results of the analysis of a number of maize oils are given in Table XXVI.

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[a] Milliau, Bertainchand, and Malet, Rapport sur les huiles d'olive de Tunisie, 1900, p. 33.

TABLE XXVI.—*Maize oil.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
De Negri and Fabris:[a]		*Degrees.*						*° C.*	*° C.*	*Per ct.*
Minimum	0.9215	....	....	84.0	....	111.1	190.0	17.0	13.0	......
Maximum	.9220	....	....	86.0	....	112.6	190.8	20.0	16.0	......
Tolman and Munson:[b]										
Minimum	.9233	76.2	1.4760	75.2	190.2	116.7	189.9	21.6	....	1.80
Maximum	.9253	77.5	1.4768	89.2	212.5	123.3	193.4	23.0	....	3.60
Archbutt[c]	.9243	....	....	81.6	....	122.7	189.7	....	....	2.40
Vulté and Gibson[d]	.9213	....	1.4766	74.0	176.0	118.6	192.6	22.4	....	1.12
Six other analysts:[e]										
Minimum	.9215	....	....	56.0	....	119.6	188.0	16.0	13.0	3.12
Maximum	.9255	....	....	89.0	....	123.9	193.4	20.0	14.0	3.50

[a] 8 samples. Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 122.
[b] 4 samples.
[c] Jour. Soc. Chem. Ind., 1899, 18: 346.
[d] Jour. Amer. Chem. Soc., 1900, 22: 453.
[e] Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 122.

The specific gravity, rise of temperature with sulphuric acid, index of refraction, and iodin absorption are much higher for maize than for olive oil, and the last three values are considerably higher than for cotton-seed oil. The high Maumené number and iodin absorption of this oil would materially influence the values of olive oil even when added in amounts as small as 10 or 15 per cent. The content of solid fatty acids of maize oil is rather low, hence the iodin number of the liquid fatty acids is not so characteristic as with cotton-seed oil. Maize oil gives a peculiar red color when shaken with nitric acid (specific gravity, 1.37), which is quite different from the color obtained with cotton-seed oil, and would serve to differentiate them.

MUSTARD-SEED OIL.

This oil belongs to the rape-seed oil group, and in general characteristics it differs but slightly from rape-seed oil, as is apparent from Table XXVII.

TABLE XXVII.—*Mustard-seed oil.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
De Negri and Fabris:[a]		*Degrees.*						° C.	° C.	*Perct.*
Minimum	0.9125	....	....	42.0	....	92.1	170.2	15.0	....	....
Maximum	.9175	....	....	45.0	....	106.5	174.6	18.0	....	....
Tolman and Munson:[b]										
Minimum	.9147	74.5	1.4750	61.4	130.9	98.4	173.0	20.8	....	0.13
Maximum	.9193	76.5	1.4762	79.4	190.3	113.0	182.8	21.5	....	1.13
Blasdale:[c]										
Minimum	.9151	....	1.4751	....	....	98.4	173.9	15.0	....	....
Maximum	.9161	....	1.4760	....	....	103.6	174.7	16.0	....	....
Crosseley and Le Sueur:[d]										
Minimum	.9142	....	1.4740	....	....	96.8	171.2	....	....	1.36
Maximum	.9155					98.8	173.3	....	....	1.85
Müntz, Durand and Milliau:[e]	.9180	....	....	39.0	108.0	96.0	....	16.0	15.0	....
Seven other analysts:[f]										
Minimum	.9142	....	....	44.0	....	96.0	....	16.0	....	....
Maximum	.9183									

[a] 6 samples. Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 37.
[b] 5 samples.
[c] 2 samples. Jour. Amer. Chem. Soc., 1895, 17: 935.
[d] 2 samples. Jour. Soc. Chem. Ind., 1898, 17: 989.
[e] 1 sample. Bulletin du Ministère de l'Agriculture, 1895, p. 139.
[f] Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 37.

It is probable that this oil is seldom if ever used as an adulterant of olive oils, although it is frequently added to rape oil, and this may find its way into olive oil. Means of detecting it in small quantities are as unsatisfactory as for rape oil, and the analyst will scarcely be able to distinguish between the oils of this group.

The presence of sulphur compounds in the oils of the *Cruciferæ* gives a means for their detection. If the oils are saponified with alcoholic potash and stirred with a silver spatula, the silver will become blackened by the formation of sulphid.

RAPE-SEED AND COLZA OILS.

Rape-seed and colza oils may be considered practically identical as regards source and composition, and for convenience the terms are considered here as being synonymous. This oil is derived from various species of *Brassica* of the family *Cruciferæ*, and according to Allen[a] is used as an adulterant for olive oil. According to the Codex Alimentarius Austriacus rape oil or colza oil is the oil from the field cabbage (*Brassica campestris*), but all the following oils are sold under the name of rape oil: Colza oil from *Brassica campestris;* rape oil from *Brassica campestris*, variety *napus;* rape oil from *Brassica campestris*, variety *rapa;* Hederich oil from *Raphanus Raphanistrum* or field radish, and radish oil from *Raphanus sativus*. Although the values of this oil would make small additions of it difficult of detection, it is not so well adapted as a substitute for olive oil as the products previously mentioned under this class, owing to the acrid taste of even the well-refined oil. Analyses of rape and colza oils are given in Table XXVIII.

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[a] Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 92.

TABLE XXVIII.—*Rape-seed and colza oils.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification number.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
		Degrees.						° C.	° C.	*Per ct.*
Crosseley and Le Sueur:[a]										
Minimum	0.9142	....	1.4744	....	....	94.1	167.7	....	....	0.36
Maximum	.9171					104.8	173.0	....	....	1.78
Tolman and Munson:[b]										
Minimum	.9143	74.1	1.4748	54.5	135.0	92.5	174.1	20.0	....	.63
Maximum	.9163	74.8	1.4752	67.8	152.5	101.2	176.6	21.9	....	3.57
Müntz, Durand, and Milliau:[c]										
Minimum	.9145	....	....	48.0	133.0	99.0	....	15.5	15.0	....
Maximum	.9150	....	....	56.0	155.0	103.0	....	17.0	16.0	....
De Negri and Fabris:[d]										
Minimum	.9150	....	....	49.0	....	97.2	175.0	16.0	....	....
Maximum	.9170	....	....	51.0	....	102.1	177.0	19.0	....	....
Twenty-one other analysts:[e]										
Minimum	.9112	....	....	50.0	....	97.0	170.0	15.5	....	....
Maximum	.9184	....	....	59.0	....	106.2	183.0	22.0	....	....

[a] 7 samples. Jour. Soc. Chem. Ind., 1898, **17**: 989.
[b] 4 samples.
[c] 2 samples. Bulletin du Ministère de l'Agriculture, 1895, p. 139.
[d] 14 samples. Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 26.
[e] Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 26.

The specific gravities of these oils are practically identical with that of the olive; the index of refraction, iodin number, and rise of temperature with sulphuric acid are considerably higher. The saponification number is much lower than for olive oil or for any of the oils used to adulterate it, with the exception of the other members of the rape-oil group. The average saponification value given for rape oil is about 173, while the average value for olive oil is about 193, and an olive oil having a saponification value much below 190 should be looked upon with suspicion. An addition of less than 20 per cent of rape oil would probably be difficult to detect, but this amount should be sufficient to materially affect the values of a normal olive oil. Low specific gravity accompanied by a high index of refraction is one of the marked characteristics of rape oil, wherein it differs from most oils which with a low specific gravity have a low refractive index.

Valenta[a] has suggested the differentiation of oils by their solubility in glacial acetic acid of 1.0562 specific gravity, and states that rape and mustard oils are the only common oils not completely soluble in this acid. Allen[a] confirms the statement of Valenta, but suggests that the solubility of an oil is greatly increased by a high percentage of free acids. The strength of acetic acid used, according to this author, may vary considerably without greatly affecting the solubility. Hurst,[b] on the other hand, finds that rape and colza oils are completely dissolved by glacial acetic acid and gives the temperature turbidity of from 73° to 99°. Thomson and Ballantyne[c] have also studied this question and find that Valenta's statement holds good only when acetic acid of specific gravity 1.0562 is employed; if an acid of specific gravity 1.0542 is used rape oil is completely soluble.

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[a] Dingler, 1884, **252**: 296; Jour. Chem. Soc., 1884, **46**: 1078.

In working in this laboratory upon Valenta's test with acid of 99.5 per cent strength it was found that rape oil and mustard oil both dissolve completely at a temperature of 120° C., although at a very slightly reduced temperature the oils are again partly precipitated. Olive oil is readily soluble at 120°, but precipitates again upon a slight reduction of temperature. Mixtures composed of 20 per cent and 40 per cent of rape oil with olive oil and similar mixtures of mustard oil showed no distinction from pure olive oil in respect to solubility. Hence as a means of detecting rape oil, and especially mixtures of the same with olive oils, this test must be considered of little value.

SUNFLOWER OIL.

Sunflower oil has a mild taste and pleasant odor, and the cold drawn oil is said to be used in some sections of Europe for culinary purposes.[d] The physical and chemical characteristics of this oil are so widely different from those of olive oil that additions of even relatively small amounts to olive oil would be sufficient to make the values of the latter abnormal. The compiled results of the analyses of sunflower oil are given in Table XXIX.

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[a] Jour. Soc. Chem. Ind., 1886, 5: 69, 282.

[b] Jour. Soc. Chem. Ind., 1887, 6: 22.

[c] Jour. Soc. Chem. Ind., 1891, **10**: 233.

[d] Jour. Soc. Chem. Ind., 1892, **11**: p. 470.

TABLE XXIX.—*Sunflower oil.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
De Negri and Fabris:[a]		*Degrees.*						° *C.*	° *C.*	*Per ct.*
Minimum	0.9260			72.0	......	119.7	188.0	22.0	18.0	
Maximum		......	......	75.0	......	120.1	189.3	24.0		......
Tolman and Munson:[b]										
Minimum	.9201	72.1	1.4736	60.0	167.0	104.1	191.2	21.0	......	0.18
Maximum	.9205	72.7	1.4739			108.3	192.3			1.72
Lewkowitsch:[c]										
Minimum	.9240	......	1.4769	67.5	......	119.7	188.0	23.0	17.0	......
Maximum	.9262			73.0	......	135.0	194.0			
Allen:[d]										
Minimum	.9240	......	......	......	......	122.0	188.4	......	......	......
Maximum	.9260	......	......	......	......	135.0	197.6	......	......	......
Seven other analysts:										
Minimum	.9260	......	......	......	......	129.0	192.0	17.0	......	......
Maximum	.9520	......	......	......	......	133.2	194.0	23.0	......	......

[a] 3 samples. Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 106.
[b] 2 samples.
[c] Oils, Fats, and Waxes. p. 356.
[d] Com. Org. Anal., 3d ed., vol 2, pt. 1, p. 93.

POPPY-SEED OIL.

According to Allen[a] and Lewkowitsch,[b] this oil is quite largely used in Europe as a salad oil and for culinary purposes, and is used somewhat as an adulterant for olive oil. In this country, however, it is not used either as a salad or cooking oil to any extent, and it is doubtful whether it is ever used as an olive-oil adulterant. From Table XXX it is seen that the specific gravity, index of refraction, Maumené number, and iodin absorption of poppy oil are exceptionally high, and these characteristics are sufficient to indicate its addition to an olive oil, although, in the absence of distinct qualitative tests, the difficulty of definitely distinguishing between this oil and some of the others already mentioned, when added in small proportions, is apparent.

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[a] Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 94.
[b] Oils, Fats, and Waxes, 2d ed., p. 352.

TABLE XXX.—*Poppy-seed oil.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
De Negri and Fabris:[a]		*Degrees.*						° *C.*	° *C.*	*Per ct.*
Minimum	0.9270	....	....	88.5	136.8	......	193.4	20.0	......	......
Maximum					137.5	......	193.8	21.0	......	......
Tolman and Munson:[b]										
Minimum	.9239	77.1	1.4766	75.8	213.0	133.2	190.2	25.4	......	0.90
Maximum	.9244	77.8	1.4770	85.5	237.5	134.9	193.8	25.8	......	2.31
Müntz, Durand, and Milliau[c]	.9250	....	....	80.0	222.0	133.0	......	20.5	16.0	......
Sixteen other analysts:										
Minimum	.9240	....	....	71.0	......	130.5	192.8	20.5		-
Maximum	.9370	....	....	86.4	......	141.0	194.0		......	......

[a] 3 samples. Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 114.
[b] 2 samples.
[c] 1 sample. Bulletin du Ministère de l'Agriculture, 1895, p. 139.

LARD OIL.

This oil is so manufactured that it remains liquid at ordinary temperatures, and when well refined is entirely free from the taste and odor of lard. When heated, however, this odor is developed, and may serve as a means of distinguishing lard oil when not mixed with other oils. If so mixed, however, even though the lard oil be largely in excess, this odor is sufficiently masked to make the test of little value. The following table gives a number of analyses of lard oil:

TABLE XXXI.—*Lard oil.*

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
Tolman and Munson:[a]		*Degrees.*						° *C.*	° *C.*	*Perct.*
Minimum	0.9148	66.8	1.4702	39.9	103.3	69.7	195.3	33.2	......	0.28
Maximum	.9175	69.5	1.4720	47.8	110.8	77.2	197.7	38.4	......	1.28
Duyk:[b]										
Minimum	.9154	65.2	1.4692	......	......	73.0	193.0	35.0	31.0	......
Maximum										
Allen:[c]										
Minimum	.9150	....	....	41.0	......	67.0	189.5	......	......	......
Maximum						82.0	196.0			
Müntz, Durand, and Milliau:[d]										
Minimum	.9160	....	....	33.0	91.0	60.0	......	35.0	34.0	......
Maximum										

[a] 4 samples.
[b] Bulletin de l'Association Belge, 1901, 15:18.
[c] Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 98.
[d] Bulletin du Ministère de l'Agriculture, 1895, p. 139.

The specific gravity, refractive index, rise of temperature with sulphuric acid, and saponification number of this oil are practically identical with the same values of olive oils. The iodin number is much lower, and the melting point of fatty acids much higher than for olive oils, but when mixed, for example, with an olive oil having a high iodin number and a consequently low melting point for its fatty acids as much as 50 to 60 per cent of the lard oil could be used without rendering these values abnormal for pure oils. The lard oils have a high content of solid fatty acids, but this factor is rendered of little value when the oil is mixed with an olive oil having a high iodin number, since such an olive oil will probably have a correspondingly low content of solid fatty acids as is shown by Table XXXIII.

MISCELLANEOUS OILS.

The oils named in tables XXXII and XXXIII were analyzed to obtain further data regarding their values, and also for the purpose of studying their use in the adulteration of olive oil. Through the kindness of the V. D. Anderson Company, of Cleveland, a number of cold-pressed oils were prepared from seeds which were identified in the seed laboratory of the Department of Agriculture. They are as follows:

Brown mustard	*Brassica arvensis* (charlock).
Black mustard	*Brassica juncea* (Indian mustard).
Yellow mustard	*Sinapis alba* (yellow mustard).
Rape	*Brassica napus.*
Poppy	*Papaver somnifera* (opium poppy).

The cocoanut oil was pressed from the flesh of cocoanuts. The peanuts used were from Virginia. The almond, cotton-seed, and linseed oils were prepared from commercial seed. Other oils were prepared in the laboratory.

There are a number of commercial oils included in these tables, but as there was no way of being assured of their purity the results obtained are not of great value. Sample No. 23606 was sent by Armour & Co. as a pure neutral lard oil and it had practically no lard odor or taste except when heated. These samples were discussed under the headings of the various oils.

TABLE XXXII.—*Description of miscellaneous oils.*

Serial No.	Kind of oil.	Source.	Description.
22077	Cocoanut	India Refining Co., Philadelphia, Pa.	Konut—cocoanut oil, lard substitute.
773	do	V. D. Anderson Co., Cleveland, Ohio.	Cold-drawn oil.
494	Palm	Schiefflein & Co., New York City	Commercial oil.
22433	Lard	Armour & Co., Chicago, Ill	Prime lard oil.
22434	do	Z. D. Gilman, Washington, D. C	
23606	do	Armour & Co., Chicago, Ill	Neutral lard oil.
487	do	Schiefflein & Co., New York City	Commercial oil.
1181	Magnolia	James Brody, Biloxi, Miss	Oil from pulp of magnolia seed.
1182	do	do	Fat from pulp of magnolia seed.
1150	do	do	Oil from pulp of magnolia seed.

TABLE XXXII.—*Description of miscellaneous oils*—Continued.

Serial No.	Kind of oil.	Source.	Description.
23656	Peanut	Z. D. Gilman, Washington, D. C.......	Commercial oil.
499	.....do	Prepared in laboratory...............	Cold-drawn oil.
13234	.....do	....	8 years old.
13235	.....do	....	8 years old; cooked oil.
772	.....do	V. D. Anderson Co., Cleveland, Ohio.	Cold-drawn oil.
1149	.....do	A. E. Leach, Boston, Mass............	
486	Mustard	Schieffelin & Co., New York City.....	Commercial oil.
495	.....do	Prepared in laboratory...............	Cold-drawn oil; black mustard.
770	.....do	V. D. Anderson Co., Cleveland, Ohio.	Cold-drawn oil; brown mustard.
771	.....do	.....do	Cold-drawn oil; black mustard.
776	.....do	.....do	Cold-drawn oil; yellow mustard.
490	Rape	Schieffelin & Co., New York City.....	Commercial oil.
496	.....do	Prepared in laboratory...............	Cold-drawn oil.
775	.....do	V. D. Anderson Co., Cleveland, Ohio.	Do.
937	.....do	V. Villavecchia, Rome, Italy..........	Colza oil di Vercelli (Picimente).
1187	Almond	V. D. Anderson Co., Cleveland, Ohio.	Cold-drawn oil.
489	Sesame	Schieffelin & Co., New York City.....	Commercial oil.
23624	Sunflower	Prepared in laboratory...............	Cold-drawn oil.
498	.....do	.....do	Do.
491	Maize	Schieffelin & Co., New York City.....	Commercial oil.
444	.....do	Glucose Sugar Refining Co., Chicago, Ill.	
497	.....do	Indianapolis Hominy Mills, Indiana.	Do.
777	.....do	V. D. Anderson Co., Cleveland, Ohio.	Cold-drawn oil.
1159	Cotton-seed ..	American Cotton Oil Co., New York City.	Commercial oil—"Butter Oil."
1160	.....do	.....do	Commercial oil—"Summer White."
1161	.....do	.....do	Commercial oil—"Cooking Oil."
1186	.....do	V. D. Anderson Co., Cleveland, Ohio.	Cold-drawn oil.
493	Poppy	Schieffelin & Co., New York City.....	
774	.....do	V. D. Anderson Co., Cleveland, Ohio.	Do.
1188	Linseed	.....do	Do.

TABLE XXXIII.—*Analyses of miscellaneous oils.*

COCOANUT OIL.

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification value.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
		Degrees.								°*C.*	*Per ct.*	*Per ct.*
22077	0.9269	....	....	....	....	7.90	....	....	....	....	....	....
773	.9259	49.1	1.4587	21.0	44.0	8.58	31.9	....	259.5	25.2	65.90	0.11

PALM OIL.

494	0.9128	....	....	....	....	53.0	99.0	....	201.0	49.2	....	19.58

LARD OIL.

22433	0.9148	67.4	1.4706	47.8	106.2	75.9	94.0	98.9	195.7	33.2	18.90	0.75
23434	.9145	67.4	1.4706	46.8	104.0	77.2	....	101.3	195.3	34.2	19.30	.78
23606	.9160	69.5	1.4720	46.5	103.3	69.7	95.8	101.3	197.7	38.4	26.68	.28
487	.9175	66.8	1.4702	39.9	110.8	72.5	93.9	97.9	196.2	35.8	21.43	1.28

MAGNOLIA OIL.

1181	0.9128	64.5	....	....	....	81.7	....	106.2	189.5	34.3	18.32	38.07
1182	....	....	....	....	....	76.1	....	106.7	189.2	38.2	24.24	48.28
1150	.9130	65.0	....	....	....	80.4	....	100.3	....	....	15.48	....

TABLE XXXIII.—*Analyses of miscellaneous oils*—Continued.

PEANUT OIL.

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification value.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
		Degrees.			•					°*C.*	*Per ct.*	*Per ct.*
23656	0.9155	67.5	1.4707	61.0	185.5	89.1	....	....	191.4	33.2	....	13.51
499	.9186	70.0	1.4723	46.5	129.1	87.8	....	....	191.8	34.3	....	[a] .40
13234	.9188	71.0	1.4729	55.5	154.2	93.4	....	....	190.7	36.0	....	[b] 2.93
[e] 13235	.9364	75.0	1.4753	67.3	186.9	77.0	....	....	199.0	37.6	....	[c] 5.96
772	.9188	71.3	1.4731	63.2	135.3	96.3	114.6	....	189.9	36.4	....	[d] .24
1149	....	70.5	1.4726	....	....	94.8	....	....	188.8	33.6	....	....
MUSTARD OIL.												
486	0.9178	76.1	1.4759	68.5	190.8	103.0	....	....	175.9	21.0	....	1.13
495	.9170	76.5	1.4762	68.2	189.4	105.8	....	115.7	176.0	21.5	4.05	.38
770	.9184	76.2	1.4760	77.6	165.4	110.4	114.2	116.8	178.5	20.8	1.06	.48
771	.9193	76.5	1.4762	79.4	169.3	113.0	119.8	121.1	182.8	20.8	2.32	.34
776	.9147	74.5	1.4750	61.4	130.9	98.4	103.1	103.0	173.0	21.1	Trace.	.13
RAPE OIL.												
490	0.9161	74.3	1.4749	54.5	151.4	92.5	100.5	96.9	175.7	20.0	0.12	3.57
496	.9143	74.3	1.4749	54.9	152.5	92.7	101.5	98.0	174.7	21.9	1.02	.63
775	.9163	74.1	1.4748	63.6	135.6	101.3	105.1	106.0	176.6	20.5	Trace.	1.26
937	.9158	74.8	1.4752	67.8	144.5	101.2	....	107.5	174.1	20.4	1.43	1.52
ALMOND OIL.												
1187	0.9186	70.9	....	45.3	117.6	96.2	....	....	192.5	23.2	....	0.48
SESAME OIL.												
489	0.9218	73.3	1.4742	61.3	170.3	106.6	....	115.4	190.7	27.4	[f] 0.70	0.44
SUNFLOWER OIL.												
23624	0.9201	72.7	1.4739	....	....	108.3	113.8	117.8	192.3	21.0	3.67	0.18
498	.9205	72.1	1.4736	60.0	166.7	104.1	105.3	113.8	191.2	21.0	4.12	1.72
MAIZE OIL.												
491	0.9233	76.2	1.4760	75.2	208.9	119.2	133.3	134.5	193.4	22.0	6.98	3.12
444	.9256	77.3	1.4767	76.5	212.5	119.7	126.6	134.8	191.5	23.0	6.70	1.80
497	....	75.6	1.4757	....	....	116.7	129.4	132.1	191.7	....	7.07	....
777	.9253	77.5	1.4768	89.2	190.2	123.3	134.5	139.8	189.9	21.6	7.44	3.66
COTTON-SEED OIL.												
1159	0.9226	72.5	1.4738	66.4	172.9	103.8	....	....	197.1	35.5	22.90	0.07
1160	.9226	72.3	1.4737	73.4	191.1	106.2	....	....	196.9	39.0	22.43	.07
1161	.9226	72.3	1.4787	66.2	172.4	104.8	....	....	196.0	39.6	23.60	.04
1186	.9236	75.6	1.4757	67.1	174.3	110.9	....	....	198.5	38.0	....	2.17

[a] 3.41 per cent arachidic acid; melting point 72.5°; cold pressed oil.
[b] 3.78 per cent arachidic acid; melting point 72.5°.
[c] 4.24 per cent arachidic acid; melting point 73°.
[d] 4.12 per cent arachidic acid; melting point 72°; cold pressed oil.
[e] Cooked oil.

TABLE XXXIII.—*Analyses of miscellaneous oils*—Continued.

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification value.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
POPPY OIL.												
		Degrees.								*°C.*	*Per. ct.*	*Per ct.*
493	0.9239	77.1	1.4766	85.5	237.5	133.2	....	....	190.2	25.4	....	2.31
774	.9244	77.8	1.4770	75.8	213.0	134.9	142.0	151.7	193.8	25.8	6.67	.90
LINSEED OIL.												
1188	0.9318	88.8	1.4831	....	....	179.5	....	....	191.7	19.2	3.88	0.40

OLIVE OILS OF KNOWN ORIGIN.

CALIFORNIA OILS.

The oils in Tables XXXIV and XXXV were obtained principally from producers of established reputations and were accompanied by affidavits as to purity. Three samples, made at the station, were obtained from G. E. Colby, of the California Agricultural Experiment Station. The oils reported on are from all parts of the State devoted to olive culture and represent practically all the different existing soils and climatic conditions, which undoubtedly exert a great influence on the chemical and physical characteristics of the samples.

The results give a good idea of the variability of California oils. The analyses are more complete than any previously published data on this subject, which gives them added value in determining the limits of the various values. The per cent of free fatty acids eliminates two oils which would otherwise give an extraordinary variation to the specific gravity and index of refraction figures, a variation that is certainly not legitimate for the grade of oils considered here. Oils such as Nos. 22619 and 673 are not fit for use as salad oils, as they contain 44 and 12 per cent of free acid respectively. This factor should always be taken into consideration in reporting low results on specific gravity and refractive index. The variety of olive and the climate and soil may greatly affect the oil; how much these factors affect the product must be decided by further study. Six samples of oil (Nos. 23456, 831, 833, 23457, 832, and 834) show the uniformity of oils produced from different varieties of olive where the soil and climatic conditions are the same and the same process of manufacture is used. The first three were made from Italian varieties and the last three from the Mission olive. The Mission olive, introduced into California by the old Mission fathers, is the variety generally grown in the State, and most of the oil made comes from it. Table XXXVI gives the compiled results of analyses of California oils.

TABLE XXXIV.—*Description of California olive oils of known origin.*

Serial No.	Source.	Description.
23692	John Bidwell estate, Chico	"Rancho Chico" pure olive oil; made from Mission olives.
23463	Fred Busby, Concord	Pure olive oil from Busby's olive grove; made from Mission olives.
22617	G. E. Colby, Berkeley	Oil from Redding Picholine olives; made in 1893.
22618	.....do	Oil from mixed varieties; made in 1898.
22619	.....do	Oil from Manzanillo olives (poor quality); made in 1900.
22713	Elwood Cooper, Santa Barbara	Highest grade olive oil; made from Mission olives.
22714	.....do	Second pressing olive oil, 10 years old; made from Mission olives.
22715	.....do	Common grade olive oil (for lubricating and similar purposes); made from Mission olives.
23458	.....do	Pure olive oil, highest grade; made from Mission olives.
23649	Ehmann Olive Co., Oroville	Pure olive oil.
844	.....do	Do.
838	C. M. Gifford, San Diego	"Gifford's Best" olive oil; made from Mission olives.
839	.....do	Olive oil just from separator; made from Mission olives.
840	.....do	Olive oil 6 weeks old; made from Mission olives.
23456	Edward E. Goodrich, Santa Clara	El Quito olive oil, Italian variety.
831	.....do	El Quito olive oil, Italian variety, 1901.
833	.....do	El Quito olive oil, Italian variety, 1902.
23457	.....do	El Quito olive oil, Mission variety.
832	.....do	El Quito olive oil, Mission variety, 1901.
834	.....do	El Quito olive oil, Mission variety, 1902.
23462	J. C. Gray, Oroville	Pure olive oil from Mount Ida olive grove; made from Picholine olives.
506	James Hill & Sons, Los Angeles	Hill's pure olive oil.
836	.....do	Hill's pure olive oil; season 1901.
837	.....do	Olive oil, first pressing, 1902.
835	.....do	Olive oil, second pressing, 1902.
1091	J. A. Kleiser, Cloverdale	Pure olive oil.
23605	Peveril Meigs, Santa Barbara	Meigs's pure olive oil; made from Mission olives.
798	.....do	Olive oil; made from Mission olives.
795	.....do	Olive oil; sample taken from top of barrel.
796	.....do	Olive oil; sample taken from middle of barrel.
797	.....do	Olive oil; sample taken from bottom of barrel.
23460	Morris & Smith, Yolo	Pure olive oil.
23461	Dr. Prosek, Guerneville	Do.
23124	J. O. Riddell, Redlands	Riddell's pure olive oil.
841	.....do	Oil from ripe olives, 1901.
842	.....do	Oil from ripe olives, 1902.
843	.....do	Oil from green olives, 1902.
23459	Vincent C. Smith, Napa	Pure olive oil; from "Glen Olive" farm.
673	.....do	Second pressing olive oil; from "Glen Olive" farm.

TABLE XXXV.—*Analyses of California olive oils of known origin.*

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification value.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
		Degrees.								° *C.*	*Per ct.*	*Per ct.*
23692	0.9168	68.5	1.4713	....	....	85.6	96.6	94.3	191.2	22.6	5.12	0.95
23463	.9164	68.4	1.4712	48.0	106.6	85.6	91.5	94.4	191.9	21.3	4.92	.79
22617	....	67.3	1.4706	....	....	78.5	....	....	194.4	30.2	....	.36
22618	....	67.5	1.4707	....	....	83.7	....	....	192.5	20.7	....	8.21
22619[a]	....	62.0	1.4672	....	....	79.6	....	....	191.8	24.5	....	44.40
22713	....	68.7	1.4715	....	....	85.3	94.9	94.3	191.1	22.4	5.11	.73
22714	....	68.5	1.4713	....	....	84.6	....	92.2	191.5	22.4	3.87	1.26
22715	....	68.2	1.4711	....	....	84.7	....	90.5	191.3	20.5	2.02	2.73
23458	.9169	68.7	1.4715	48.4	107.5	88.2	94.9	96.8	191.4	23.5	4.42	.73
23649	.9164	68.0	1.4710	43.5	96.6	83.4	88.9	....	192.1	22.6	....	1.43
844	.9180	68.5	1.4713	46.9	97.9	84.2	....	....	189.7	25.8	....	.35
838	.9169	69.2	1.4718	49.5	108.5	86.2	....	96.4	190.6	24.0	6.15	1.07
839	.9171	69.2	1.4718	48.4	106.0	89.0	....	98.2	189.9	21.2	4.94	3.51
840	.9169	68.9	1.4716	48.2	105.7	86.3	....	96.3	189.4	24.0	5.94	1.09
23456	.9169	68.3	1.4711	47.7	106.0	84.3	93.4	95.4	191.9	23.4	7.23	.53
831	.9168	68.8	1.4715	46.6	98.1	85.2	....	....	189.3	20.6	....	.61
833	.9170	68.8	1.4715	48.0	101.0	84.8	....	95.2	189.8	20.2	6.55	.65
23457	.9166	68.3	1.4711	47.0	104.4	86.2	91.2	93.5	191.6	21.1	3.39	.97
832	.9178	68.8	1.4715	47.6	100.1	84.9	....	93.2	189.3	20.5	5.40	.85
834	.9168	68.8	1.4715	46.2	97.2	84.5	....	94.4	189.3	19.4	6.03	.34

[a] Not included in average on account of high percentage of free acid.

TABLE XXXV.—*Analyses of California olive oils of known origin*—Continued.

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification value.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
		Degrees.								° C.	*Per ct.*	*Per ct.*
23462	.9162	66.9	1.4708	48.0	95.5	79.9	91.7	94.4	192.2	31.0	10.91	.79
506	.9174	68.5	1.4713	38.0	98.9	84.4	....	....	193.1	22.4	....	.71
886	.9174	68.8	1.4715	48.2	105.7	86.0	....	....	190.7	24.8	....	2.51
837	.9167	68.4	1.4713	45.0	98.6	84.2	....	93.1	190.3	23.6	5.16	.51
885	.9167	68.2	1.4711	44.8	98.2	82.7	....	92.6	190.4	26.0	6.19	.96
1091	.9167	68.8	1.4715	45.8	94.5	81.9	....	....	190.0	26.6	12.96	1.42
23605	.9171	68.5	1.4718	47.1	104.2	88.5	94.5	95.0	191.5	20.2	2.43	1.54
798	.9177	68.8	1.4715	51.0	107.3	89.7	....	....	190.0	19.2	....	.63
795	.9177	69.0	1.4717	50.0	105.2	89.7	....	98.7	189.8	19.8	4.73	.75
796	.9177	68.8	1.4715	52.1	109.7	89.8	....	98.8	189.9	19.2	4.69	.61
797	.9177	68.8	1.4715	50.2	105.6	89.8	....	98.6	189.7	19.6	5.44	.63
23460	.9167	68.2	1.4711	47.0	104.0	85.7	93.8	95.8	190.4	23.4	6.24	2.24
23461	.9162	67.5	1.4707	45.0	100.0	83.0	92.4	95.4	192.0	28.0	7.62	1.07
23124	.9171	68.2	1.4711	45.5	....	83.7	92.5	93.7	193.3	25.4	6.20	.47
841	.9168	68.8	1.4715	47.1	98.3	86.9	....	....	189.4	21.6	8.61	.20
842	.9168	68.7	1.4715	46.8	97.7	87.2	....	....	189.5	21.4	7.52	.21
843	.9169	68.6	1.4714	45.6	95.2	85.1	....	....	189.6	22.8	4.33	.28
23459	.9162	67.7	1.4709	45.5	101.2	82.9	90.3	93.3	191.6	25.0	5.69	1.72
673[a] ...	.9149	66.4	1.4699	45.5	95.0	83.3	....	....	189.5	21.6	7.58	12.11
Average	.9170	68.4	1.4713	46.9	101.8	85.3	92.8	95.0	190.9	22.9	5.86	1.20
Max	.9180	69.2	1.4718	52.1	109.7	89.8	96.6	98.8	194.4	31.0	12.96	44.40
Min	.9162	62.0	1.4703	38.0	94.5	78.5	88.9	90.5	189.3	19.2	2.02	.20

[a] Not included in average on account of high percentage of free acid.

TABLE XXXVI.—*Analyses of California olive oils.* (Compiled.)

Analysts.	Determinations.								
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Free fatty acids as oleic.
Blasdale:[a]		*Degrees.*						° C.	*Per ct.*
Minimum	0.9161	....	1.4710	45.0	....	80.0	190.5	21.0	......
Maximum	.9174	....	1.4716	47.0	....	86.5	193.5	26.0	......
Tolman and Munson:[b]									
Minimum	.9162	66.9	1.4703	38.0	94.5	78.5	189.3	19.2	0.20
Maximum	.9180	69.2	1.4718	52.1	109.7	89.9	194.6	31.0	44.40
Colby:[c]									
Minimum	.9140	....	1.4689	......	....	77.7	187.0	21.0	......
Maximum	.9185	....	1.4717	......	....	93.5	193.5	26.0	......

[a] 11 samples. Jour. Amer. Chem. Soc., 1895, 17: 935.
[b] 42 samples.
[c] California Agr. Expt. Sta. Rept., 1897–98, p. 165.

ITALIAN OLIVE OILS.

These oils, results upon which are given in Tables XXXVII and XXXVIII, were obtained through V. Villavecchia, chemist of the custom-house at Rome, and Giacomo Dellapiane fu Andrea, of Genoa. The oils represent all the large oil-producing districts of Italy. It is impossible to give the variety of olive used on account of the great number of varieties in Italy; each small district may have a distinct variety of olives. The Italian olive oils are not materially different from the California oils. They have a somewhat lower Hübl number, and the percentage of solid fatty acids and the melting point of the free fatty acids are on the average considerably higher. One noticeable difference between the two oils was observed after they had stood in an ice box for several weeks. Many of the California oils showed no separation of solid fats and none of them became solid, while nearly all the Italian oils did become solid. This characteristic also held with the commercial oils. The French oils resembled in this respect the Italian oils. Tables XXXIX and XL give the compiled results of analyses of Italian and miscellaneous olive oils, respectively.

TABLE XXXVII.—*Description of Italian olive oils of known origin.*

Serial No.	Source.	Description.
580.....	Fred Baller & Co., Messina...........	Pure olive oil (Bari).
933.....	V. Villavecchia, Rome...............	Olio di oliva di Bari (Puglie).
960.....	Giacomo Dellepiane fu Andrea, Genoa.	Bari, first quality.
961.....	.....do	Bitonto.
935.....	V. Villavecchia, Rome................	Olio di oliva di Bitonto (Puglie).
936.....	.....do	Olio di oliva di Monopoli (Puglie).
962.....	Giacomo Dellepiane fu Andrea, Genoa.	Molfetta, Bari.
931.....	V. Villavecchia, Rome................	Olio di oliva di Toscana.
932.....	.....do	Finest sublime Lucca oil, S. Rae & Co.
958.....	Giacomo Dellepiane fu Andrea, Genoa.	Lucca (Toscane); Lucca, first grade.
959.....	.....do	Lucca (Toscane); Lucca, second grade.
934.....	V. Villavecchia, Rome...............	Olio di oliva di Liguria (Genoa).
952.....	Giacomo Dellepiane fu Andrea, Genoa.	Maurizio (Liguria), Monte della Grasia.
953.....	.....do	Genoa (Liguria), Spezia.
954.....	.....do	Genoa (Liguria), Genoa.
955.....	.....do	Olio fino, Genoa (Liguria), Loano.
957.....	.....do	Olio superfine, Genoa (Liguria), Loano.
956.....	.....do	Abruzzo, Fossacesia.

TABLE XXXVIII.—*Analyses of Italian olive oils of known origin.*

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification value.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
		Degrees.								°C.	*Pr. ct.*	*Pr. ct.*
580..............	0.9177	67.9	1.4709	39.6	103.1	81.8	......	....	191.7	28.5	......	......
938..............	.9158	67.7	1.4708	43.5	95.6	80.4	94.8	101.8	192.0	26.2	16.47	1.02
960..............	.9159	67.6	1.4707	44.7	101.8	80.9	96.0	102.9	191.4	26.6	16.87	.80
961..............	.9162	67.6	1.4707	43.5	95.6	80.8	96.5	98.7	191.7	27.3	13.67	.69
935..............	.9162	67.7	1.4708	44.2	97.1	80.5	95.1	100.3	191.9	29.3	15.20	1.03
936..............	.9155	67.7	1.4708	44.8	98.4	81.8	96.2	96.8	191.5	26.2	11.07	2.79
962..............	.9159	67.6	1.4707	43.5	99.1	81.2	96.5	104.1	191.8	27.9	17.72	.62
931..............	.9166	67.6	1.4708	44.4	97.4	80.7	89.8	89.1	189.6	25.8	5.01	.82
932..............	.9167	67.5	1.4707	44.4	97.4	80.5	......	....	190.1	25.2	8.07	.80
958..............	.9158	68.0	1.4710	48.0	102.3	81.8	90.6	91.4	190.4	22.9	6.04	.79
959..............	.9164	68.0	1.4710	46.2	98.4	81.5	92.0	93.0	190.5	23.9	7.95	.63
934..............	.9159	67.6	1.4707	42.5	96.8	81.1	92.4	93.2	190.6	24.5	8.60	.92
952..............	.9160	68.0	1.4710	45.0	97.9	81.4	90.5	92.2	190.1	21.6	7.38	.61
953..............	.9161	67.3	1.4705	47.0	100.0	79.2	90.8	92.2	191.3	24.9	9.76	1.18
954..............	.9180	68.5	1.4713	48.3	103.0	86.1	97.5	98.0	190.5	25.0	7.76	2.55
955..............	.9157	67.8	1.4709	41.2	97.1	82.6	94.8	95.3	190.8	23.5	8.93	1.09
957..............	.9166	68.0	1.4710	45.0	97.9	82.1	91.1	91.2	189.7	21.6	5.53	1.90
956..............	.9164	68.3	1.4712	49.1	104.7	84.5	98.1	103.1	190.7	28.5	13.51	.57
Average...	.9163	67.8	1.4709	44.9	99.1	81.6	91.0	96.5	190.9	25.5	10.50	1.11
Maximum.	.9180	68.5	1.4713	49.1	104.7	86.1	98.4	104.1	192.0	29.3	17.72	2.79
Minimum .	.9155	67.3	1.4705	39.6	95.6	79.2	89.8	89.1	189.6	21.6	5.01	.57

TABLE XXXIX.—*Italian olive oils.* (*Compiled.*)

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
De Negri and Fabris: [a]		*Degrees.*						° *C.*	° *C.*	*Pr. ct.*
Minimum	[b]0.9160			32.0		79.1	187.9			
Maximum	.9180			36.5		88.3	192.2			
De Negri and Fabris: [a]										
Minimum	[c].9145			33.0		81.1	185.0			
Maximum	.9178			36.5		89.8	192.0			
De Negri and Fabris: [a]										
Minimum	[d].9160			32.0		79.0	188.8			
Maximum	.9180			37.0		87.1	192.3			
Average	.9166					83.7				
Müntz, Durand and Milliau: [e]										
Minimum	.9158			30.0	83.3	83.7		24.0	22.0	
Maximum	.9163			35.0	97.2	84.0		24.5	23.0	
Tortelli and Ruggeri [f]	.9169			37.0				23.7		
Tolman and Munson: [g]										
Minimum	.9155	67.3	1.4703	39.6	95.6	79.2	189.7	21.6		0.61
Maximum	.9180	68.5	1.4713	49.1	104.7	86.1	192.0	29.3		2.79
Maximum	.9180				104.7	89.8	192.0	29.3		
Minimum	.9145				83.3	79.0	185.0	24.0		

[a] Annali del Laboratorio chimico centrale delle Gabelle, vol. 2, Gli Olii, pt. 2, p. 114.
[b] 18 samples. Oil from green olives.
[c] 17 samples. Oil from ripe olives.
[d] 53 samples. Pure oils from various sources.
[e] 4 samples. Bulletin du Ministère de l'Agriculture, 1895.
[f] Annali del Laboratorio chimico centrale delle Gabelle, 1900, vol. 4, p. 249.
[g] 18 samples.

TABLE XL.—*Miscellaneous olive oils.* (*Compiled.*)

Analysts.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
Müntz, Durand and Milliau (oils from Africa): [a]		*Degrees.*						° *C.*	° *C.*	*Per ct.*
Minimum	0.9169			30.0	83.3	84.1		24.0	22.5	
Maximum	.9172			35.0	97.2	84.5		26.0	24.5	
Müntz, Durand and Milliau (oils from Spain): [b]										
Minimum	.9160			30.0	83.3	83.8		24.0	22.0	
Maximum				35.0	97.2					
Müntz, Durand and Milliau (oils from Portugal): [b]										
Minimum	.9167			30.0	83.3	84.1		24.5	23.5	
Maximum				35.0	97.2					
Müntz, Durand and Milliau (oils from Greece): [b]										
Minimum	.9160			30.0	83.3	84.3		25.0	23.5	
Maximum				35.0	97.2					
Müntz, Durand and Milliau (oils from Turkey) [b]:										
Minimum	.9162			30.0	83.3	84.1		24.5	23.0	
Maximum				35.0	97.2					
Müntz, Durand and Milliau (oils from the Levant): [b]										
Minimum	.9165			30.0	83.3	83.5		24.5	23.0	
Maximum				35.0	97.2					

[a] 4 samples. Bulletin du Ministère de l'Agriculture, 1895, p. 139.
[b] Bulletin du Ministère de l'Agriculture, 1895, p. 139.

TABLE XL.—*Miscellaneous olive oils*—Continued.

Analysis.	Determinations.									
	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Saponification value.	Melting point of fatty acids.	Solidifying point of fatty acids.	Free fatty acids as oleic.
Müntz, Durand and Milliau (oils from Tunis): [a]		*Degrees.*						° C.	° C.	*Perct.*
Minimum	.9150	......	......	......	......	81.4	......	24.0	22.5	......
Maximum	.9182	......	......	......	......	85.2	......	27.0	25.0	......
Milliau, Bertainchand and Malet (oils from Tunis) [b]:										
Minimum	.9170	......	......	30.0	84.0	79.3	......	24.8	20.2	0.60
Maximum	.9196	......	......	36.0	97.0	89.5	......	38.0	31.9	7.6
Average	.9183	......	......	......	......	85.0	......	32.0	......	1.4
Müntz, Durand and Milliau (oils from France): [c]										
Minimum	.9154	......	......	30.0	83.3	83.5	......	24.0	22.5	......
Maximum	.9168	......	......	35.0	97.2	84.1	......	25.0	24.0	......
Tolman and Munson (oils from Montevideo): [d]	......	68.9	1.4716	......	......	79.2	194.6	26.8	......	1.40
Tolman and Munson (oils from San Antonio): [d]	......	67.8	1.4710	......	......	83.4	190.7	23.4	......	4.33

[a] 15 samples. Bulletin du Ministère de l'Agriculture, 1895, p. 139.

[b] 49 samples. Bulletin de l'Agriculture et Commerce de Tunis.

[c] 7 samples. Bulletin du Ministère de l'Agriculture, 1895, p. 139.

[d] These two South American oils were exhibited at the World's Fair, Chicago. From W. C. Blasdale, Berkeley, Cal.

COMMERCIAL OLIVE OILS.

One hundred and fifty-seven samples of commercial olive oils were examined, of which 72 were labeled French oils, 67 were Italian oils, 15 were California oils, and the remaining 3 were of miscellaneous origin. Sixty-one samples were furnished by the Treasury Department and came from the appraisers' stores at New York and Philadelphia; 96 samples were bought upon the markets at Boston, Buffalo, Chicago, New Haven, New Orleans, St. Louis, San Francisco, Oakland, and Washington. The following table summarizes the results obtained on these commercial oils:

TABLE XLI.—*Summary of commercial oils.*

Kind and source.	Total number of samples.	Samples containing oils other than olive.	Samples containing cotton-seed oil.
French oils:			
From appraiser's stores	33	3	0
Purchased in market	39	8	5
Italian oils:			
From appraiser's stores	28	2	0
Purchased in market	39	8	6
Total imported oils:			
From appraiser's stores	61	5	0
Purchased in market	78	16	11
California oils, purchased in market	15	2	2
Miscellaneous oils,[a] purchased in market	3	3	2

[a] One sample sold as "superior to any olive oil" was maize oil.

Descriptions of the commercial samples are given in Tables XLII, XLIV, XLVI, and XLVIII, and the results of the analyses are given in Tables XLIII, XLV, XLVII, and XLIX. Table XLIII gives the results obtained on the commercial samples of California oils. These oils agree very closely with the California oils of known purity given in Table XXXV. The melting point of the fatty acids of No. 23114 is rather high, but the percentage of solid fatty acids is also high, and the iodin number is correspondingly low.

TABLE XLII.—*Description of commercial California oils not found adulterated.*

Serial No.	Source.	Capacity of receptacle.	Price.	Price of 10 ounces.	Label.
		Ounces.	*Dollars.*	*Dollars.*	
22432	C. C. Bryan, Washington, D. C.	20.3	1.00	0.49	California Olive Oil, Olivina. Julius Paul Smith, Livermore, Cal.
22616	F. J. Lea & Co., Oakland, Cal.	....	....	....	
23069	Alameda Co., Boston, Mass.	15.5	.60	.38	California Olive Oil. Alameda Co., Boston, Mass. Absolutely Pure.
23099	Sunset Grocery Co., Oakland, Cal.	25.3	1.00	.39	Absolutely Pure. "San Pedro."
23101	Torrey & Gardiner, Oakland, Cal.	19.6	1.00	.51	Old Mission Olive Oil. Akerman & Tuffley, San Diego, Cal.
23109	A. Simon, San Francisco, Cal.	....	.90	....	Coburn's Olive Oil. Coburn, Tevis & Co., San Francisco, Cal.
23112	John Schloen, San Francisco, Cal.	19.2	1.00	.52	Gifford's Best California Olive Oil. Absolutely Pure. C. M. Gifford, Jamacha Valley, San Diego Co., Cal.
23114	L. Lebenbaum & Co., San Francisco, Cal.	20.3	1.85	.45	Santa Ana, California, Olive Oil. First Pressing.
23116	J. Caire Co., San Francisco, Cal.	22.0	1.00	.41	Pure Olive Oil. Quito Olive Farm, Santa Clara Co., Cal.
23117	.....do	20.9	1.00	.47	Pure Olive Oil. Pala Grove, Alum Rock, San Jose, Cal.
23118	.....do	19.6	1.00	.51	Pure Olive Oil. Olivina Farm, Auburn, Cal. Mrs. Emily Roberson.
23124	J. O. Riddel, Redlands, Cal.	....	....	....	Riddel's Olive Oil. First Pressing. J. O. Riddel, Redlands, Cal.
23125	.....do	....	....	....	Pure Olive Oil. F. M. Hunt, Redlands, Cal.

TABLE XLIII.—*Analyses of commercial California oils not found adulterated.*

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Hübl number.	Iodin number of liquid fatty acids.		Saponification value.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
						Determined.	Calculated.				
		Degrees.							*° C.*	*Per ct.*	*Per ct.*
22432	0.9152	68.7	1.4715	41.0	84.5	....	91.5	191.6	22.5	3.22	1.27
22616	....	67.4	1.4707	....	81.0	....	....	192.6	25.0	....	3.49
23069	.9157	67.1	1.4705	43.0	79.0	....	89.8	193.4	26.2	7.45	....
23099	.9163	67.7	1.4708	43.5	81.6	88.4	85.9	190.5	21.9	4.01	.29
23101	.9177	67.9	1.4710	51.0	83.8	93.9	94.9	192.9	26.1	7.18	3.96
23109	.9163	67.9	1.4710	44.5	80.3	88.5	92.0	193.2	25.8	8.32	1.18
23112	....	68.1	1.4711	46.3	79.9	....	93.3	192.4	25.2	7.96	....
23114	.9167	67.4	1.4707	49.5	79.9	94.9	97.0	194.9	33.4	13.20	.76
23116	.9171	68.9	1.4717	45.0	83.4	90.9	94.1	191.9	22.4	5.47	.69
23117	.9167	68.6	1.4714	43.8	83.7	89.4	92.1	192.2	20.7	3.62	.80
23118	.9164	68.0	1.4710	45.2	81.0	88.7	90.1	193.1	23.1	5.62	1.24
23124	.9171	68.2	1.4711	45.5	83.7	92.5	93.7	193.3	25.4	6.20	.47
23125	....	67.9	1.4710	48.0	86.5	93.2	95.0	193.2	23.2	5.42	7.25
Average...	.9165	68.0	1.4710	45.5	82.2	91.2	92.5	192.7	24.7	6.47	1.95
Maximum.	.9177	68.9	1.4717	51.0	86.5	94.9	97.0	194.9	33.4	13.20	3.96
Minimum.	.9152	67.1	1.4705	41.0	79.0	88.4	85.9	190.5	20.7	3.22	.29

The following letter was received in regard to the analytical results obtained on sample No. 23117:

OAKLAND, CAL., *April 28, 1903.*

In returning the accompanying statement of the chemical analysis of Pala Grove oil I would like to make one change. Just before receiving the paper from Washington I had a test made of this year's product by F. M. Curtis & Son, analytical chemists in San Francisco. They returned me the following:

Specific gravity at 15.5° C	0.9147
Free fatty acids (in terms of oleic) ... per cent..	0.36
Iodin number (Hübl)	83.9
Cotton-seed oil	None.

You will note the difference in the percentage of free fatty acids, your analysis calling for 0.80 per cent. I was at a loss to account for the great difference until I recalled the age of the oil you had tested. The Justinian Caire Company have had no oil of ours since the 1901 crop, and when that was put in still had some of the 1900 crop on hand, so that what you bought from them was *at least* 2 years old.

Thanking you for your courtesy, I am, very respectfully,

CARRIE CUTLER MCLENEGAN.

PALA OLIVE GROVE, *San Jose, Cal.*

Tables XLIV and XLV give the description of the commercial French oils and the results obtained thereon. Tables XLVI and XLVII give the same data for commercial Italian oils. Like the Italian oils of known purity, the commercial French and Italian oils have a lower iodin value, a higher percentage of solid fatty acids, and the fatty acids have a higher melting point than the California oils.

TABLE XLIV.—*Description of commercial French olive oils not found adulterated.*

Serial No.	Source.	Capacity of receptacle.	Price.	Price of 10 ounces.	Label.
		Ounces.	*Dollars.*	*Dollars.*	
7049	Cobb, Bates, Yerxa & Co., Boston, Mass.	....	....	....	Huile d'Olive Vierge, d'Aix, Dupont & Cie., Bordeaux, France.
20943	Appraiser's office, Port of Philadelphia.	9.8	....	....	Vidheau et Cie., Bordeaux.
20955	A. M. & J. Solari, New Orleans, La.	17.6	0.65	0.37	Huile d'Olive Vierge, J. L. Duret & Co., Bordeaux, France.
20956	.....do	19.6	.45	.23	Huile d'Olive Surfine, Adolphe Puget, Marseille.
20957	.....do	19.6	.40	.20	Huile d'Olive, Superfine Raffinée, James Plagniol, Marseille.
21003	J. A. Kochl, New Orleans, La.	18.6	.65	.35	V. S. O. Huile d'Olive, Extra Superfine, J. de Brie & Co., Bordeaux, France.
21063	J. G. Swarbuck, New Orleans, La.	9.12	.40	.44	Huile d'Olive Vierge, 9 oz. Qualité Supérieure, Larronde Frères, Bordeaux, France.
21071	S. W. Clark & Son, New Orleans, La.	20.2	.65	.32	Huile d'Olive, Extra Surfine, Talbot Frères, Bordeaux, France.
21072	.....do	20.2	.65	.32	Nicelle Olive Oil, Seville Packing Co., New York. Made in Nice, France.
21112	Appraiser's office, Port of Philadelphia.	20.9	....	....	Huile d'Olive, Superfine, A. de Luze & Fils, Bordeaux.
21397	.....do	20.0	....	....	Huile d'Olive, Superfine Extra. Packed expressly for I. J. Miller, 21st and Brandywine streets, Philadelphia, Pa.
21398	.....do	21.3	....	....	Huile d'Olive, Extra Surfine, Bordeaux. Bottled for Newton, Robertson & Co., Hartford, Conn.
21399	.....do	....	....	....	Huile d'Olive, Garneau Frères, Bordeaux.
21870	Appraiser's office, Port of New York.	16.9	....	....	Huile d'Olive, Superfine Clarifiée, Barton & Guestier, Bordeaux.
21871	.....do	17.2	....	....	Huile d'Olive Vierge, Qualité extra supérieure, Beaumarchand Fils, Bordeaux.
21872	.....do	....	....	....	
21873	.....do	....	....	....	

TABLE XLIV.—*Description of commercial French olive oils not found adulterated*—C't'd.

Serial No.	Source.	Capacity of receptacle.	Price.	Price of 10 ounces.	Label.
		Ounces.	*Dollars.*	*Dollars.*	
21874	Appraiser's office, Port of New York.	....	....	....	
21875	.....do	23.3	....	....	Huile d'Olive, Joseph Mayrargue, Nice, France.
21876	.....do	17.2	....	....	Huile d'Olive, Superfine Clarifiée, Roulan Frères, Bordeaux.
21877	.....do	16.9	....	....	Huile d'Olive, Qualité Supérieure, F. P. Garrettson & Co., New York City.
21878	.....do	....	....	....	
21879	.....do	....	....	....	
21880	.....do	....	....	....	
21881	.....do	17.5	....	....	Huile d'Olive Vierge, Extra, James Plagniol, France.
21882	.....do	18.5	....	....	Sublime Olive Oil. Imported by W. P. Allen, Philadelphia, Pa.
21883	.....do	....	....	....	
21884	.....do	....	....	....	
21885	.....do	....	....	....	
21886	.....do	....	....	....	
21887	.....do	....	....	....	
21888	.....do	27.4	....	....	Huile d'Olive, Boutelleau Fils. Made in France.
21889	.....do	....	....	....	
21890	.....do	....	....	....	
21891	.....do	....	....	....	
21892	.....do	16.9	....	....	Huile d'Olive, Superfine, Jules Gaston & Cie., Bordeaux.
21893	.....do	....	....	....	
21894	.....do	....	....	....	
22908	Faxon, Williams & Faxon, Buffalo, N.Y.	17.9	0.70	0.39	Huile d'Olive Vierge bottled in Bordeaux, for Faxon, Williams & Faxon.
22913	John A. Seel, Rochester, N. Y.	22.0	.68	.31	Pure Olive Oil packed in Bordeaux for J. A. Seel.
22914	.....do	8.7	.40	.46	Huile d'Olive, Barton & Guestier, Bordeaux.
22918	E. C. Cochrane Co., Buffalo, N. Y.	30.4	.75	.25	Vierge Extra Huile d'Olive, E. C. Cochrane Co.
22919	.....do	17.5	.85	.49	Huile d'Olive, Brandenburg Frères, Bordeaux.
22920	Dingens Bros., Buffalo, N. Y.	....	....	....	Huile d'Olive Vierge, J. E. Blanc, Marseille.
23066	S. S. Pierce, Boston, Mass.	32.1	1.25	.39	Huile d'Olive Vierge, V^{ve} Chaffard, Jardin de la France.
23071	Cobb, Aldrich & Co., Boston, Mass.	8.7	.35	.40	Huile d'Olive de Nice, J. P. Fourché, Nice.
23088	R. N. Nesbit Co., New Haven, Conn.	16.9	.70	.41	Huile d'Olive, A. Fontenelle Fils & Cie., Grasse.
23092	E. E. Nichols, New Haven, Conn.	16.9	.80	.47	Huile d'Olive Laselle, Chapnelle & Cie., Aix.
23103	M. D. Myer, San Francisco, Cal.	15.2	.75	.49	Huile d'Olive, De Possel Fils, Marseille.
23105	Goldberg, Bowen & Co., San Francisco, Cal.	18.9	.65	.34	Huile d'Olive Vierge, bottled in Bordeaux.
23110	A. Liebmann, San Francisco, Cal.	23.0	.70	.30	Choicest Imported Olive Oil, bottled by A. Liebmann.
23111	Bebo, Newman & Ikenberg, San Francisco, Cal.	18.6	.75	.40	Huile d'Olive Vierge, Alphonse Billet, Bordeaux.
23128	L. Stumpf Grocery Co., St. Louis, Mo.	23.6	.95	.40	Huile d'Olive Vierge de Provence, L. A. Price, Bordeaux.
23129	Connor, Eagan Co., St. Louis, Mo.	17.9	.80	.44	Huile d'Olive, J. Mottet & Cie., Grasse.
23133	H. L. Eppling, Chicago, Ill.	20.6	.75	.36	Club House Brand, Virgin Olive Oil, Grasse. Imported by Franklin MacVeagh & Co., Chicago.
23135	S. G. Anable, Chicago, Ill.	20.3	1.00	.49	Huile d'Olive Vierge, Alexis Godillot Jeune.
23136	Whiteman & Co., Chicago, Ill.	21.3	.90	.40	Huile d'Olive, Supérieure, Dumouron & Cie., Grasse.
23138	Hamelsfaler, Chicago, Ill.	16.2	.85	.52	Huile d'Olive Vierge, Alphonse Pinard, Bordeaux.
23139	C. Jevne & Co., Chicago, Ill.	19.2	1.00	.52	Huile d'Olive, St. Benedictus. Imported specially for Christian Jevne & Co., Chicago.
23140	.....do	21.3	.75	.35	Huile d'Olive, Société Hygiénique Alimentaire.
23141	.....do	24.3	.75	.30	Importé spécialement par C. Jevne & Co.
23083	Gilbert & Thompson, New Haven, Conn.	31.4	1.40	.44	Club Brand, Virgin Salad Oil. Imported by G. F. Heublein & Bro., Hartford, Conn.

TABLE XLV.—*Analyses of commercial samples of French olive oils not found adulterated.*

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification value.	Melting points of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
		Degrees.								° *C.*	*Per ct.*	*Per ct.*
7049	0.9183	67.5	1.4707	....	....	85.0	....	....	....	....	....	....
20943	.9180	68.1	1.4711	43.2	96.0	80.3	91.5	....	195.2	30.2	....	1.32
20955	.9157	68.2	1.4711	42.0	93.2	79.8	90.6	....	193.4	26.2	....	1.32
20956	.9165	67.4	1.4707	45.2	100.3	83.0	97.4	....	192.6	30.6	....	1.56
20957	.9173	67.9	1.4709	48.8	108.4	81.4	....	98.3	193.2	27.8	12.69	1.02
21003	.9164	68.0	1.4710	41.8	92.8	79.8	91.5	....	192.7	25.2	....	1.32
21063	....	67.4	1.4707	40.7	90.4	80.5	95.5	92.2	192.3	25.2	8.21	2.62
21071	.9161	67.8	1.4709	43.6	96.9	79.0	....	....	194.1	26.0	....	1.91
21072	....	67.5	1.4707	42.0	98.3	80.9	89.5	88.1	192.3	25.2	3.74	.76
21112	....	67.8	1.4709	47.2	104.9	81.2	90.0	....	193.5	30.6	....	1.56
21397	.9163	67.3	1.4706	42.0	93.2	81.3	93.2	95.1	192.8	25.0	10.14	1.52
21398	.9167	67.0	1.4704	43.8	97.3	80.8	94.1	98.5	193.1	29.4	13.56	1.20
21399	.9169	67.5	1.4707	43.8	97.3	81.4	93.9	97.7	193.0	25.8	12.20	2.16
21870	.9164	67.6	1.4707	45.2	100.3	80.7	91.2	....	192.8	29.0	....	2.29
21871	.9163	68.0	1.4710	43.8	97.3	79.5	91.6	94.7	194.4	....	11.62	3.63
21872	.9165	67.3	1.4706	43.0	95.5	79.9	93.2	93.8	192.6	27.0	10.44	.92
21873	.9167	68.0	1.4710	46.5	103.3	81.8	99.4	97.8	193.5	27.0	11.94	.66
21874	.9166	67.6	1.4708	47.2	104.9	82.9	94.6	....	195.0	28.2	....	1.55
21875	.9163	67.8	1.4709	44.4	98.6	81.4	92.6	93.9	191.6	26.7	8.92	.96
21876	.9163	67.5	1.4707	44.4	98.6	79.7	94.9	96.0	192.8	....	12.50	2.75
21877	.9166	68.1	1.4711	44.6	99.1	80.0	96.0	....	194.8	29.4	....	2.00
21878	.9166	67.8	1.4709	45.0	100.0	79.0	....	97.4	195.3	28.0	14.46	2.39
21879	....	68.0	1.4711	47.6	105.8	84.2	100.0	97.5	192.6	28.4	9.26	1.55
21880	.9172	68.5	1.4713	47.4	105.3	83.1	98.4	100.0	193.5	27.0	12.38	.45
21881	.9165	68.0	1.4710	41.4	92.0	80.1	....	96.3	194.5	28.2	12.36	.71
21882	.9174	68.0	1.4710	47.4	105.3	80.8	92.7	99.7	194.0	30.4	14.45	.90
21883	.9167	68.2	1.4711	47.4	105.3	83.8	......	99.7	192.1	25.2	11.56	.52
21884	.9170	68.1	1.4710	42.4	94.2	83.7	93.1	93.8	193.5	27.2	6.33	1.76
21885	....	68.2	1.4711	44.4	98.6	80.2	95.7	95.7	193.6	28.0	11.93	.66
21886	.9165	67.8	1.4709	42.8	95.1	80.9	....	94.1	194.2	27.2	9.58	.89
21887	.9153	67.6	1.4707	45.5	101.1	81.2	....	92.6	192.1	28.8	7.89	1.34
21888	.9173	67.5	1.4707	42.4	94.2	80.1	....	98.1	194.3	24.6	13.95	1.09
21889	.9163	67.7	1.4707	42.1	93.5	80.8	96.0	94.3	195.3	26.7	9.92	.47
21890	....	68.0	1.4711	44.0	97.8	82.8	93.7	94.3	193.6	26.9	7.73	.89
21891	....	67.8	1.4709	43.7	97.1	82.5	93.2	94.6	192.7	27.4	8.34	.84
21892	.9163	68.0	1.4710	40.7	90.4	79.8	91.3	....	195.1	28.0	....	2.14
21893	.9163	67.5	1.4707	41.4	92.0	81.5	....	91.1	191.7	23.7	6.17	.48
21894	....	67.5	1.4707	42.6	94.6	81.1	....	....	194.2	24.7	....	3.44
22908	....	68.7	1.4709	42.2	93.7	80.9	....	91.2	193.5	24.2	6.81	3.18
22913	....	67.2	1.4704	42.8	95.5	80.2	90.6	89.7	193.2	25.2	6.11	1.83
22914	.9153	67.3	1.4705	45.5	101.1	83.6	....	....	193.1	27.6	....	2.58
22918	.9152	67.8	1.4709	42.4	94.2	80.9	....	....	191.2	26.3	....	2.28
22919	.9157	68.0	1.4711	42.5	94.4	81.9	93.1	94.6	193.6	27.2	9.06	1.98
22920	.9167	66.4	1.4699	51.5	114.4	83.6	92.1	....	191.7	27.2	....	1.82
23066	.9161	67.2	1.4705	48.8	108.4	82.5	....	94.8	192.6	25.0	8.56	1.06
23071	.9170	67.1	1.4705	47.2	104.9	80.6	....	91.1	191.7	27.5	7.11	2.93
23088	.9167	67.6	1.4708	44.0	97.8	80.6	....	89.5	192.8	25.0	5.53	1.73
23092	.9177	67.4	1.4706	51.4	114.2	80.5	....	96.2	194.2	30.0	15.95	1.07
23103	.9196	67.4	1.4706	47.6	105.8	82.5	91.1	97.9	193.0	29.6	11.37	2.06
23105	.9168	67.8	1.4708	48.2	107.1	80.8	93.4	94.2	191.3	28.6	9.87	1.38
23110	.9168	67.7	1.4709	49.4	109.7	84.2	....	101.8	193.0	30.8	12.81	1.66
23111	.9173	67.8	1.4708	50.0	111.1	80.4	94.7	97.3	193.1	29.4	12.90	.81
23128	.9166	67.6	1.4707	48.2	107.1	81.4	92.8	93.5	192.2	27.2	8.50	1.62
23129	.9160	67.8	1.4709	46.0	102.2	82.2	....	92.8	192.1	29.0	6.99	.95
23133	.9177	67.5	1.4707	44.0	97.8	80.5	....	89.1	190.5	23.8	5.25	.90
23135	.9170	67.7	1.4708	46.0	102.4	81.0	....	95.7	191.5	28.0	10.90	.99
23136	.9158	67.2	1.4705	45.4	100.8	79.4	....	91.3	192.3	27.8	8.59	2.27
23138	.9150	67.6	1.4707	45.4	100.8	82.2	....	....	193.3	26.3	....	1.69
23139	.9165	67.6	1.4707	49.5	110.0	81.4	97.9	97.1	192.0	29.6	11.70	1.71
23140	.9164	67.6	1.4707	46.5	103.3	80.5	94.7	93.0	191.3	28.1	8.98	2.22
23141	.9172	67.2	1.4705	48.0	106.6	80.1	92.3	93.7	191.3	27.7	10.00	3.31
Av ..	.9166	67.7	1.4708	45.1	100.1	81.3	93.7	94.9	193.0	27.3	9.98	1.59
Max.	.9196	68.7	1.4713	51.5	114.4	85.0	100.0	101.8	195.3	30.8	15.95	3.63
Min .	.9150	66.4	1.4699	40.7	90.4	79.0	89.5	88.1	190.5	23.7	3.74	.45

TABLE XLVI.—*Description of Italian olive oils not found adulterated.*

Serial No.	Source.	Capacity of receptacle.	Price.	Price of 10 ounces.	Label.
		Ounces.	*Dollars.*	*Dollars.*	
21907	Appraiser's office, Port of New York.				Bari.
21911	do				Bari.
22910	John A. Seel, Rochester, N. Y.	131.0	2.50	0.19	Olio d'Oliva Finissimo, Garantito Genuino, A. Nicolini, Bari.
21895	Appraiser's office, Port of New York.				Sorrento.
21908	do				Naples.
21915	do				Naples.
23082	Santo Capasso, New Haven, Conn.	71.0	1.00	.14	Olio d'Oliva Finissimo, Faicchio & S. Lorenzello, Prov. di Benevento.
20958	A. M. & J. Solari, New Orleans, La.	16.2	.55	.34	Olio d'Oliva di Lucca, F. Bertolli.
21111	Appraiser's office, Port of Philadelphia.				Olio d'Oliva Suprafino, F. Bertolli, Lucca.
21199	do	8.4			Lucca Finest Cream Salad Oil, Warranted Pure Olive Oil. Smith, Kline & French Co., Philadelphia.
21896	Appraiser's office, Port of New York.				Lucca.
21897	do				Lucca.
21898	do				Lucca.
21899	do				Lucca.
21902	do				Lucca.
21909	do				Lucca.
23068	S. S. Pierce, Boston, Mass.	10.1	.35	.34	Pure and Best Cream Olive Oil.
23093	Booth Meat Co., New Haven, Conn.	18.6	.75	.40	Cream Olive Oil, Made in Leghorn for W. A. Castle, Springfield, Mass.
23095	Bronson & Platt Co., New Haven, Conn.	8.7	.35	.40	Huile d'Olive Excelsior, Toscana Excelsior, Lucca.
23097	H. Olson, Oakland, Cal.	23.6	.65	.27	Pure Lucca Oil, Crosse & Blackwell, London.
23100	Agard & Russell, Oakland, Cal.	24.7	.85	.34	Extra Sublime Cream Lucca Olive Oil.
23106	John Bulotti, San Francisco, Cal.	25.4	.65	.25	Fine Lucca Oil, Imported by John Bulotti, San Francisco, Cal
23107	H. Weeden, San Francisco, Cal.	13.5	.50	.37	The Best Lucca Olive Oil, G. Fontana & Co., San Francisco, Cal.
23113	Goldberg, Bowen & Co., San Francisco, Cal.	19.9	.60	.30	Sublime Lucca Oil, Goldberg, Bowen & Co., San Francisco, Cal.
23122	Mrs. C. Goessel, San Francisco, Cal.	21.3	.65	.30	Genuine Italian Olive Oil, M. Ricci & Co., Lucca.
23123	H. Glander, San Francisco, Cal.	23.6	.60	.25	Pure Lucca Olive Oil, Robert Baldocchi & Co., San Francisco, Cal.
21900	Appraiser's office, Port of New York.				Leghorn.
21901	do				Leghorn.
21905	do				Leghorn.
21910	do				Leghorn.
21912	do				Leghorn.
21917	do				Leghorn.
21918	do				Leghorn.
22907	Faxon, Williams & Faxon, Buffalo, N. Y.	19.2	.70	.36	Finest Sublime Lucca Olive Oil, S. Rae & Co., Leghorn.
22911	John A. Seel, Rochester, N. Y.	68.1	1.50	.22	Sublime Italian Lucca Olive Oil, G. Bartoni, Leghorn.
22912	John A. Seel, Rochester, N. Y.	23.6	1.25	.52	The Routh Extra Sublime Lucca Olive Oil, H. L. Routh & Sons.
22915	E. M. Buehl, Buffalo, N. Y.	16.9			Extra Cream Lucca Olive Oil, D. G. Rossetti & Co., Leghorn.
22921	Dingens Bros., Buffalo, N. Y.	10.1	.65	.64	Pure Genuine Olive Oil, Augustus Achiardi, Leghorn.
22922	do				A. Nicolini.
23094	Booth Meat Co., New Haven, Conn.	12.5	.35	.28	Huile d'Olive Lucca, Nicoli Pitito, Leghorn.
23104	L. M. Walter, San Francisco, Cal.	27.1	1.00	.36	Olio Soprafino, Fratelli Alfonso, Leghorn, Lucca.
23132	Schlieswohl & Peterson, Chicago, Ill.	19.0	.70	.36	Finest Sublime Lucca Oil, S. Rae & Co., Leghorn.
23134	Geo. P. Popf, Chicago, Ill.	18.9	.75	.39	Extra Superfine Italian Salad Oil, Warranted Pure Olive Oil, Antonini & Co., Leghorn.
23074	Conway Co., Boston, Mass.	18.6	.75	.40	Olio d'Oliva di Lucca, Ditta Ferd. Nencioni, Pisa. B. Ferrari & Co., Boston, sole importers.
23073	The Ginter Grocery Co., Boston, Mass.	18.2	.60	.33	Pure Tuscan Olive Oil, William Lloyd & Co., Leghorn.
21903	Appraiser's office, Port of New York.				Genoa.

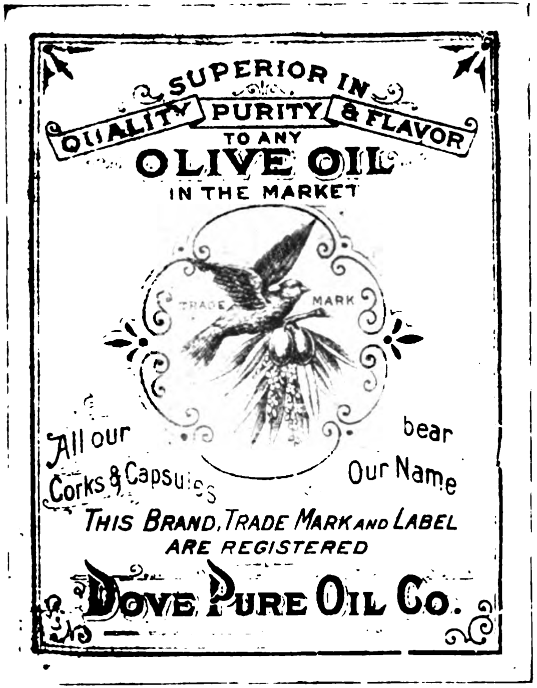


FIG. 1.—LABEL ON BOTTLE OF MAIZE OIL.
(SEE PAGE 5.)

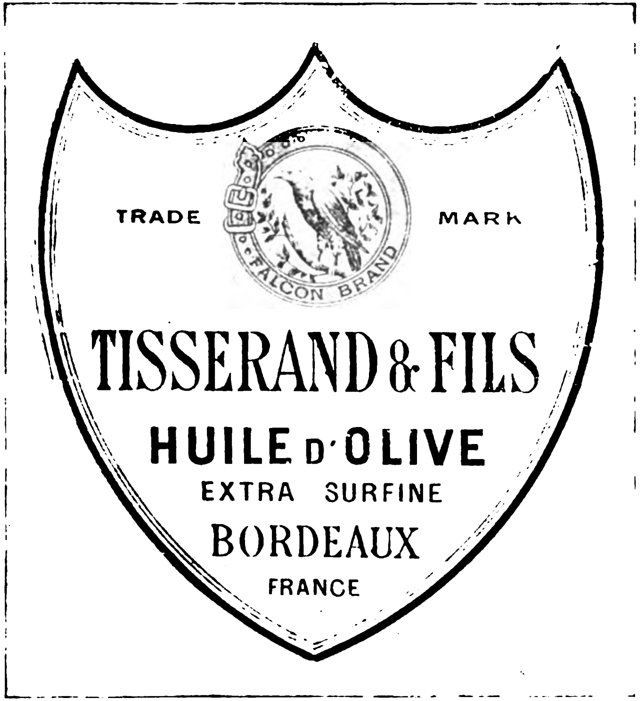
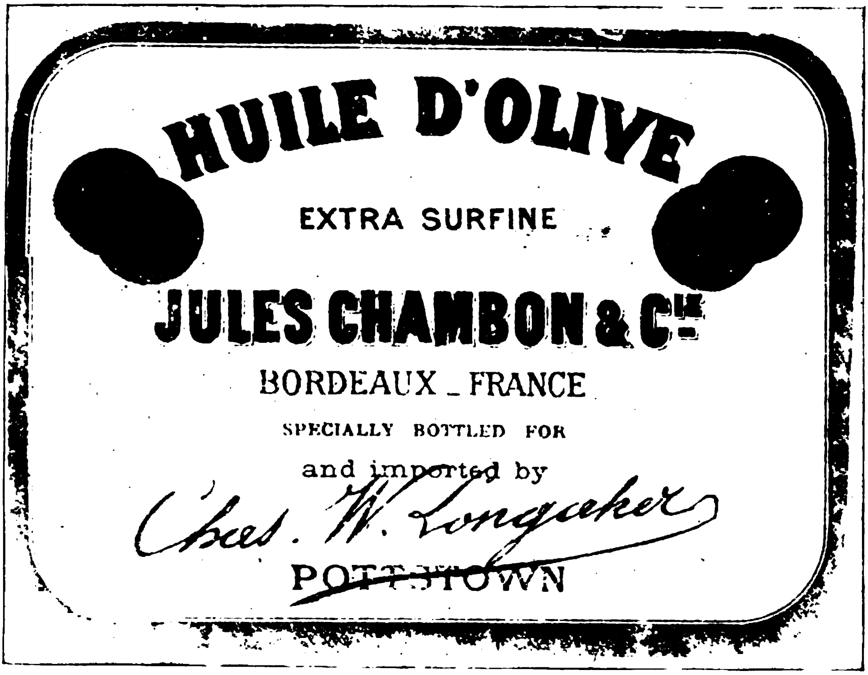
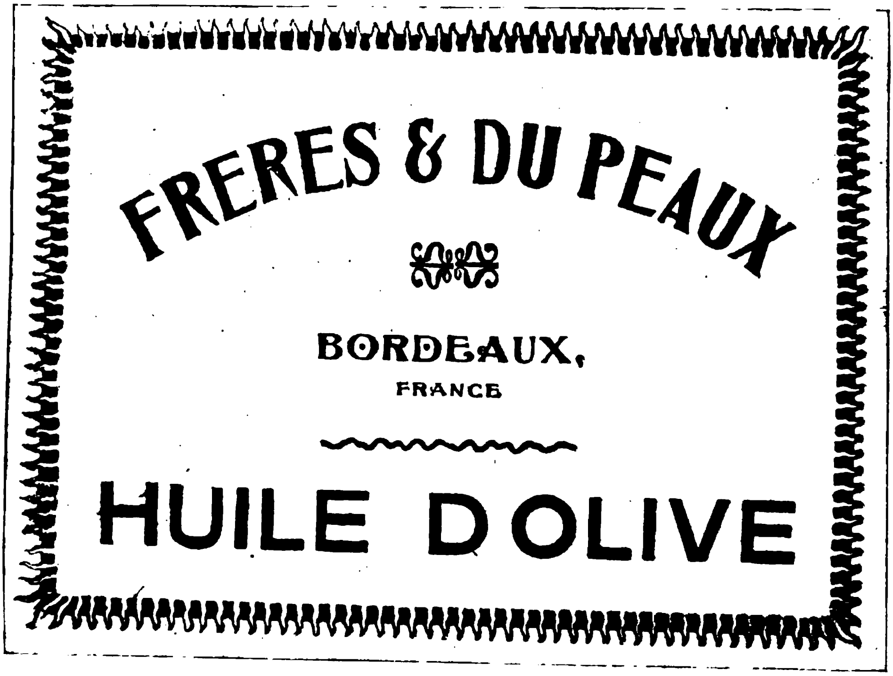


FIG. 2.—LABEL ON OLIVE OIL MIXED WITH COTTON-SEED OIL.
(SEE PAGES 4 AND 60.)



HUILE D'OLIVE
EXTRA SURFINE
JULES CHAMBON & Cie
BORDEAUX _ FRANCE
SPECIALLY BOTTLED FOR
and imported by
Chas. W. Longacker
POTTSTOWN

FIG. 1.—LABEL ON OLIVE OIL MIXED WITH PEANUT OIL. (SEE PAGES 5 AND 60.)



FRERES & DU PEAUX
BORDEAUX,
FRANCE
HUILE D OLIVE

FIG. 2.—LABEL ON BOTTLE OF COTTON-SEED OIL. (SEE PAGES 4 AND 60.)

TABLE XLVI.—*Description of Italian olive oils not found adulterated*—Continued.

Serial No.	Source.	Capacity of receptacle.	Price.	Price of 10 ounces.	Label.
		Ounces.	*Dollars.*	*Dollars.*	
21904...	Appraiser's office, Port of New York.	....	....	....	Genoa.
21913...	.....do	....	....	....	Genoa.
21916...	.....do	....	....	....	Genoa.
21919...	.....do	....	....	....	Genoa.
23119...	Smith Cash Store, San Francisco, Cal.	17.1	.60	.35	Olio Vero d'Oliva Pio Moro fu Corngliano, Liguria.
22909...	A. Z. Giannelli, Buffalo, N. Y.	30.4	1.00	.32	Pure Olive Oil, A. Z. Giannelli, Casciana d'Monsagrati.
23067...	S. S. Pierce, Boston, Mass.	21.9	.65	.30	Pure Olive Oil, Italy, S. S. Pierce & Co., Boston, Mass.
23084...	E. E. Hall & Sons, New Haven, Conn.	30.4	.90	.29	Italian Olive Oil, Edw. E. Hall & Son, New Haven, Conn.
23187...	V. Kaufman, Chicago, Ill.	....	.75	....	Collinfiore Royal Table Olive Oil, Italy.
21029...	Popovich & Abramovich, New Orleans, La.	128.0	1.90	.15	Giov. di Cola, Termini, Sicily.
21914...	Appraiser's office, Port of New York.	....	....	....	Messina.

TABLE XLVII.—*Analyses of commercial samples of Italian olive oils not found adulterated.*

Serial No.	Specific gravity at 15.5° C.	Butyrorefractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification number.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
		Degrees.								°*C.*	*Per ct.*	*Per ct.*
21907	0.9171	67.7	1.4708	42.1	93.6	81.2	93.7	97.5	193.3	....	12.28	2.22
21911	9152	67.2	1.4704	41.9	93.1	81.1	....	98.1	193.5	....	12.95	4.56
22910	.9165	67.0	1.4704	47.0	104.4	82.4	91.9	....	194.2	28.4	....	4.61
21895	.9167	67.1	1.4703	42.0	93.3	77.9	90.6	94.8	193.7	28.4	13.42	2.72
21908	....	67.8	1.4709	42.2	93.7	80.9	91.2	94.0	193.5	25.2	9.46	3.18
21915	.9134	66.2	1.4698	47.6	105.8	82.4	....	94.1	194.1	26.7	8.04	15.25
23082	.9161	67.4	1.4706	47.7	106.0	82.2	....	....	193.4	29.6	....	4.40
20958	.9159	67.5	1.4707	40.0	88.9	79.7	....	89.6	193.5	24.0	6.57	1.69
21111	.9163	67.4	1.4707	42.0	93.4	80.9	92.5	....	192.6	27.2	....	1.06
21199	....	67.5	1.4707	41.4	92.0	81.2	90.0	....	192.2	25.9	....	1.53
21896	....	67.6	1.4708	44.0	97.7	82.4	....	....	196.6	28.2	....	1.18
21897	.9159	67.2	1.4705	40.5	90.0	79.2	90.8	....	194.5	27.7	....	.78
21898	.9167	67.8	1.4709	46.0	102.0	81.4	92.7	94.8	192.6	28.3	9.66	1.83
21899	.9163	67.3	1.4705	42.6	94.6	81.2	91.4	93.9	193.0	29.2	9.16	2.16
21902	.9154	67.9	1.4710	42.7	94.8	82.8	98.6	96.3	193.1	27.2	9.58	3.32
21909	....	67.2	1.4704	41.4	92.0	81.4	....	92.0	192.3	....	7.16	1.75
23068	.9166	67.9	1.4705	45.0	100.0	80.1	93.8	91.9	192.1	28.9	8.50	2.63
23093	.9161	67.2	1.4705	48.8	108.4	79.9	....	88.8	192.7	22.8	5.58	1.07
23095	.9166	68.3	1.4712	48.5	107 7	82.1	94.2	....	192.4	28.5	....	.93
23097	.9153	67.1	1.4705	43.5	96.6	79.6	90.0	90.6	191.5	26.9	7.69	1.51
23100	.9158	67.1	1.4705	48.2	107.1	82.0	91.7	....	192.6	23.8	....	4.28
23106	.9156	67.2	1.4705	45.4	100.9	80.9	....	90.0	191.8	25.0	6.80	1.29
23107	.9152	67.1	1.4704	46.8	104.0	80.5	....	90.1	191.1	22.6	6.25	3.17
23113	.9155	67.0	1.4704	45.0	100.0	79.4	90.4	91.2	191.3	27.8	8.50	2.33
23122	.9167	67.3	1.4706	47.7	106.0	81.4	....	93.3	191.1	26.3	8.29	3.09
23123	.9165	67.6	1.4708	44.0	97.6	80.1	91.8	90.7	191.2	24.6	7.22	1.29
21900	....	67.5	1.4706	41.2	91.5	80.9	91.3	92.1	192.5	25.8	7.69	1.74
21901	....	67.2	1.4704	41.4	92.0	80.6	....	91.0	193.8	26.8	6.97	1.76
21905	....	67.8	1.4709	41.2	91.5	80.7	....	....	192.2	27.9	....	2.12
21910	.9166	67.3	1.4705	39.8	88.4	80.9	91.1	91.5	191.6	25.6	7.14	1.61
21912	.9173	67.5	1.4706	44.0	97.8	81.6	94.7	94.9	192.2	28.0	9.54	2.90
21917	....	67.4	1.4705	42.6	94.6	80.9	....	91.6	193.8	28.3	7.23	1.93
21918	.9152	67.8	1.4709	42.4	94.2	81.9	....	....	....	....	....	2.28
22907	.9157	66.7	1.4701	45.0	100.0	80.1	91.0	88.0	191.9	23.8	5.44	1.06
22911	.9155	67.5	1.4707	43.6	96.9	82.5	....	....	192.8	24.3	....	1.31
22912	.9163	67.4	1.4706	43.0	95.5	80.1	....	....	191.2	25.1	....	1.26
22915	.9157	66.9	1.4702	45.0	100.0	80.1	92.4	91.1	192.2	28.6	7.60	1.74
22921	....	67.1	1.4704	43.0	95.5	80.2	90.1	90.4	190.1	26.1	6.80	2.15
22922	.9170	67.6	1.4708	46.0	102.2	79.7	93.3	....	194.0	28.0	....	3.89
23094	.9169	67.6	1.4708	45.9	102.0	79.2	90.2	....	191.6	26.2	....	1.65
23104	.9166	67.6	1.4708	46.0	102.2	80.6	93.0	91.6	190.5	25.1	7.53	1.16
23132	.9163	67.3	1.4706	44.0	97.6	79.3	....	89.8	190.2	26.3	7.28	.90
23134	.9159	67.5	1.4707	44.5	98.8	79.2	93.0	90.9	190.9	27.1	8.28	.98

TABLE XLVII.—*Analyses of commercial samples of Italian olive oils not found adulterated*—Continued.

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer reading at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of liquid fatty acids.		Saponification number.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.
							Determined.	Calculated.				
		Degrees.								°*C.*	*Per ct.*	*Per ct.*
23074....	.9165	66.8	1.4701	45.9	102.0	81.2	....	88.4	191.6	24.4	3.70	2.46
23073....	.9175	67.1	1.4705	41.4	92.0	78.5	....	90.0	191.9	21.0	8.52	1.22
21903....	.9150	67.3	1.4705	41.4	92.0	80.6	91.9	95.1	192.5	29.4	10.74	3.87
21904....	.9162	67.8	1.4709	44.2	98.2	81.6	....	94.8	192.4	29.4	9.47	1.59
21913....	.9163	67.2	1.4704	42.8	95.1	80.2	90.6	95.2	193.2	25.2	11.26	1.83
21916....	.9179	67.6	1.4707	44.7	99.3	81.5	92.7	98.5	194.6	30.4	12.68	2.09
21919....	.9157	68.0	1.4711	42.5	94.4	84.5	99.7	100.2	193.6	27.2	11.21	1.98
23119....	.9156	66.9	1.4703	47.5	105.5	82.6	93.3	....	193.5	28.0	....	5.80
22909....	.9154	66.8	1.4701	44.0	97.8	82.7	....	90.8	192.7	26.0	4.57	2.27
23067....	.9159	66.9	1.4702	44.4	98.6	80.8	....	92.8	192.1	25.6	8.50	2.63
23084....	.9165	67.4	1.4706	48.4	107.5	83.3	....	94.4	192.6	25.0	7.38	.72
23137....	.9154	68.0	1.4710	44.2	98.2	82.5	....	90.5	193.7	24.2	4.40	1.60
21029....	....	66.3	1.4699	40.8	90.7	77.5	91.9	91.9	190.9	26.2	11.22	3.61
21914....	.9153	67.3	1.4705	45.5	101.1	83.6	94.5	....	193.1	30.4	....	2.58
Av..	.9161	67.3	1.4706	44.0	97.8	80.9	92.4	92.61	192.6	26.6	8.35	2.42
Max.	.9179	68.3	1.4712	48.8	108.4	84.5	99.7	100.2	196.6	30.4	13.42	15.25
Min.	.9134	66.2	1.4698	39.8	88.4	77.5	90.0	88.0	190.1	21.0	3.70	.72

Tables XLVIII and XLIX give the data obtained upon the samples of commercial oils found to be adulterated. The samples containing peanut oil all bore French labels and those containing sesame oil Italian labels. Two California oils contained cotton-seed oil. In all of the above cases the oils were represented to be pure olive oil and in most cases were guaranteed pure and of special grade. One of the most striking facts brought out by this examination of imported oils received from the customs officers, and therefore, known to be imported, was that none of them was adulterated with cotton-seed oil.

TABLE XLVIII.—*Description of adulterated olive oils.*

Serial. No.	Source.	Capacity of receptacle.	Price.	Price of 10 ounces.	Label.
		Ounces.	*Dollars.*	*Dollars.*	
21027...	A. L. Bühler, New Orleans, La.	7.4	0.10	0.13	Huile d'Olive Vierge. E. Loubon, Nice, France.
21031...	Popovich and Abramovich, New Orleans, La.	59.5	.90	.17	Olio d'Oliva. Egisto Dini, Lucca, Italy.
21110...	Appraiser's office, port of Philadelphia.	....	....	....	Sublime Olive Oil. Addisoni Fils, Masina, Italy.
21113...	.....do	....	....	....	Huile d'Olive, Extra Surfine. Jules Chambon & Cie., Bordeaux, France. Imported by Chas. W. Longaker, Pottstown.
21395...	.....do	....	....	....	Huile d'Olive. P. M. Loubric, Bordeaux, France. Packed for I. K. Bean.
21396...	.....do	....	....	....	Huile d'Olive. P. M. Loubric, Bordeaux, France. Imported for J. M. Oliver & Sons.
21906...	.....do	....	....	....	Lucca, Italy.
22924...	Dingens Bros., Buffalo, N. Y,	67.0	1.40	.20	Olio Sopraffino. Umberto Albertini, Livorno, Italy.
22925...	.....do	120.1	2.25	.19	Olio Puro d'Oliva. Restivo & Co., Lucca, (Tuscana), Italy.
23070...	Rice Bros., West Newton, Mass.	....	.35	....	Huile d'Olive, Vierge. Silas Peirce & Co., Bordeaux, France.

TABLE XLVIII.—*Description of adulterated olive oils*—Continued.

Serial No.	Source.	Capacity of receptacle.	Price.	Price of 10 ounces.	Label.
		Ounces.	*Dollars.*	*Dollars.*	
23072...	Cobb, Bates, Yerxa & Co., Boston, Mass.[a]	16	0.55	0.34	Huile d'Olive Vierge, d'Aix. Dupont & Cie., Bordeaux, France.
23085...	E. E. Hall & Son, New Haven, Conn.	11.5	.59	.51	Huile d'Olive, Extra Vierge. Naegely & Pasero, Marseille, France.
23086...	.....do	11.5	.45	.39	Huile d'Olive, Vierge, d'Aix. Alex. Eyquem, Bordeaux, France.
23087...	S. Francisconi, New Haven, Conn.	34.1	.50	.14	Olio d'Oliva. Luigi di Cos Matteucci, Lucca, Italy.
23090...	G. Savarese, New Haven, Conn.	29.7	.50	.15	Olio d'Oliva, Sopraffino. F. Berio & Co., Lucca, Tuscana, Italy.
23096...	D. M. Welch & Son, New Haven, Conn.	8.7	.30	.34	Huile d'Olive, Extra Surfine. Tisserand & Fils, Bordeaux, France.
23108...	F. W. Ruchers & Co., San Francisco, Cal.	18.6	.50	.26	Olio d'Oliva, Sopraffino. Gaetano Giurlani, Lucca, Italy.
23120...	H. C. Kätterhorn, San Francisco, Cal.	25.3	.65	.25	Olio d'Oliva Vergine. De Martini E Cia., Riviera di Genova, Italy.
23121...	S. Scatena, San Francisco, Cal.	24.3	.50	.20	Olio Vergine Purissimo. Garantito di Lucca. S. Scatena & Co.
23131...	Luyties Bros., St. Louis, Mo.	....	.25	....	Huile d'Olive, Clarifée. Martinot Frères, Bordeaux, France.
23089...	D. Dove, New Haven, Conn.[b]	4.1	.10	.25	Huile Salad. Giacomo Luigi, Nove Orleons, Americano.
21030...	Popovich & Abramovich, New Orleans, La.	....	1.00	....	T. A. Fucich & Son, Lussinpiccolo, Austria.
22916...	Boston Store, Buffalo, N. Y.	11.1	.12	.18	Huile d'Olive. Frères & Du Peaux, Bordeaux, France.
22917...	.....do	....	.43	....	Superior in Quality, Purity, and Flavor to any Olive Oil in the market. Dove Pure Oil Co.
23098...	Howland & Co., Oakland, Cal.[c]	20.2	.60	.30	Olive Oil. El Montecito Man'f'g. Co., El Montecito, Cal.
23102...	W. P. Wheeler, Oakland, Cal.[c]	24.7	.65	.26	"Ramona" Pure Olive Oil. W. P. Wheeler, Oakland, Cal.

[a] On being notified that this oil contained peanut oil, Cobb, Bates & Yerxa replied, "We wish to say to you that we buy the Dupont oil to be not only pure olive oil but the best to be had." A second sample of a different shipment, secured by our representative from Cobb, Bates & Yerxa without their knowledge, was found to be pure. For analysis of pure sample, No. 7049, see page 55.

[b] A letter addressed to Giacomo Luigi, New Orleans, La., was returned unopened.

[c] See correspondence given below.

The following letters were received in regard to the two brands of California oil found to be adulterated. The second letter needs no comment, but the first brings out a very important fact, namely, the use or misuse of labels of well-known brands to cover gross adulterations.

MONTECITO, CAL., *May 7, 1903.*

Yours of 14th ultimo, with analysis of "El Montecito Manufacturing Company's Olive Oil," at hand.

In reply, I beg to state that the above company was organized in 1893 to make a *really pure* olive oil, and it never put out or sold a drop of any but pure olive oil.

The making of oil was discontinued in 1896 and the last of the stock on hand was sold in August, 1899, to Haas Brothers, of San Francisco, some 1,400 bottles. The machinery was sold last year, and the fine stone building costing nearly $7,000 is for sale. *Pure* olive oil could not be sold in competition with adulterated spurious oils, and as the parties interested in this company would not resort to such methods, the company simply went out of business. The company never sold any oil to Howland & Co., of Oakland.

Respectfully, yours,

EL MONTECITO M'F'G CO.,
EDWIN H. SAWYER *President.*

OAKLAND, CAL., *May 6, 1903.*

Your favor under date of April 14, 1903, at hand and contents noted. Beg to say in reply that I am much surprised to learn result of your analysis of my "Ramona" brand of olive oil, as when I adopted this brand for my retail trade I had the oil thoroughly analyzed, with result showing freedom from any adulterants. I presumed that the same brand of oil from the same producers and importers would run same in quality and purity, but it seems, as you remark in your favor referred to before, that "manufacturers vary in the goods they turn out." It must be so in this case, and I shall certainly change the oil and obtain a *pure* oil.

Permit me to say that I am not a wholesaler, simply bottling oil for my *retail* business. It seems, on my part, to be simply a case of too much trust imposed in manufacturers' agents.

I would remark that parties to whom I have submitted a sample of oil, now bottled under brand mentioned, for analysis, report adulteration of about 5 per cent. However, that would suffice to necessitate its being discarded as a pure article.

I beg to remain, yours, truly,

W. P. WHEELER.

The Department of State, upon the request of the Secretary of Agriculture, instructed the United States consul at Bordeaux to investigate the existence of the firms named on the labels reproduced on Plates I and II as manufacturers of olive oil at that port. The following excerpts are made from the report rendered by the consul:

1. No such firms as Tisserand & Fils, or Frères & Du Peaux, or Jules Chambon & Cie are known in this consulate or appear on the books thereof during the year 1903.
2. No such firms are to be found in the last directory of the city of Bordeaux.

Whether there are such firms in Bordeaux can be conclusively determined only by an examination of the registry books, for which a fee of 35 centimes (7 cents) per volume is charged for each volume included in the search. * * *

It is well-nigh impossible that there should be such a firm as "Frères & Du Peaux." It might be "Dupeaux Frères" or "Dupeaux & Frères." * * *

From all I can gather I think there is somewhere in Bordeaux a firm which sometimes calls itself "Jules Chambon & Cie," but where it is, who compose it, or what is its business the most searching inquiry has failed to reveal. Of the other two firms named not even a trace or suspicion can be found.

I am, sir, very respectfully, your obedient servant,

ALBION W. TOURGEE,

Consul of the United States of America at Bordeaux, France.

JULY 30, 1903.

TABLE XLIX.—*Analyses of adulterated olive oils.*

Serial No.	Specific gravity at 15.5° C.	Butyro-refractometer readings at 15.5° C.	Index of refraction at 15.5° C.	Maumené number.	Specific temperature reaction.	Hübl number.	Iodin number of free fatty acids.		Saponification value.	Melting point of fatty acids.	Solid fatty acids.	Free fatty acids as oleic.	Halphen test.	Renard test.	Baudouin test.	Adulterant.
							Determined.	Calculated.								
		Degrees.								°*C.*	*Per ct.*	*Per ct.*				
21027	....	72.8	1.4740	77.8	172.9	107.2	142.0	....	195.8	37.2	....	0.30	Positive			Cotton-seed oil
21030	0.9207	70.7	1.4727	67.8	150.6	93.3	....	....	194.8	35.7	....	1.78	do			Do.
21031	.9228	72.3	1.4737	70.2	156.0	94.9	....	....	197.0	37.2	....	2.25	do			Do.
21110	.9166	68.0	1.4710	42.4	94.2	85.2	97.5	96.7	191.4	26.0	7.40	1.50	Negative	Negative	Positive	Sesame oil.
21113	.9150	67.6	1.4708	45.2	100.3	81.1	98.0	94.9	192.2	26.7	10.15	1.98	do	Positive	Negative	Peanut oil.
21395	.9166	67.4	1.4707	43.4	96.4	80.7	98.2	....	191.7	25.2	10.83	1.63	do	do	do	Do.
21396	.9169	67.3	1.4706	41.8	92.9	81.0	92.1	94.7	192.4	27.0	9.96	2.04	do	do	do	Do.
21906	.9173	67.9	1.4710	45.7	101.5	81.1	92.1	95.7	193.2	26.0	10.78	1.88	do		Positive	Sesame oil.
22924	.9213	71.0	1.4729	....	....	102.4	136.7	....	196.5	37.8	....	1.40	Positive	Negative	Negative	Cotton-seed oil.
22916	.9225	72.9	1.4740	....	....	105.3	....	136.3	194.4	37.9	17.87	.07	do			Do.
22917	.9218	75.5	1.4756	....	....	107.7	117.3	121.4	191.4	24.2	6.87	.20	Negative	Negative	Negative	Maize oil.
22925	.9215	73.3	1.4742	....	....	107.4	133.0	....	195.5	37.6	....	1.16	Positive	do	Negative	Cotton-seed oil.
23070	.9170	67.9	1.4709	49.2	109.3	82.7	....	96.3	191.4	24.5	9.73	1.60	Positive (weak)	Positive	Slight, 1 to 2 per cent.	Peanut oil.
23072	.9159	67.2	1.4705	46.2	102.6	81.4	....	94.8	191.9	29.0	9.68	1.43	Negative	do	Negative	Do.
23085	.9200	69.9	1.4722	67.5	150.0	90.1	....	....	195.9	31.0	....	.40	Positive			Cotton-seed oil.
23086	.9159	68.0	1.4710	52.5	116.6	82.5	....	98.2	190.8	27.0	11.47	1.49	Negative	Positive	Negative	Peanut oil.
23087	.9167	68.1	1.4710	45.2	100.4	82.9	93.0	100.0	193.8	28.0	12.60	2.17	do	Negative	Positive	Sesame oil.
23089	.9228	73.0	1.4741	....	....	108.3	139.9	141.5	194.3	38.2	19.04	.05	Positive			Cotton-seed oil.
23090	.9162	67.9	1.4709	48.0	106.6	82.3	95.4	97.6	192.7	25.4	11.27	2.22	Negative		Positive	Sesame oil.
23096	.9181	68.8	1.4715	55.2	122.6	84.2	92.2	....	192.0	27.9	....	1.67	Positive			Cotton-seed oil.
23108	.9198	70.0	1.4723	69.5	154.4	93.8	104.5	113.1	192.8	32.1	12.60	1.08	do			Do,
23120	.9162	67.3	1.4706	47.0	104.4	80.6	....	....	190.7	27.8	....	3.20	Positive, slight, 2 per cent.			Do.
23121	.9196	70.5	1.4726	69.0	153.4	97.4	....	117.7	193.4	34.0	12.78	2.34	Positive			Do.
23131	.9227	72.7	1.4740	....	....	108.8	139.6	....	196.5	38.0	....	.16	do			Do.
23098	.9229	73.0	1.4741	....	....	109.9	137.4	....	194.3	38.0	....	.09	do			Do.
23102	.9195	70.1	1.4724	67.0	148.9	92.4	106.5	113.2	195.2	34.6	13.90	1.20	do			Do.

SUMMARY.

(1) The olive oil consumed in this country is largely imported from France and Italy. The amount produced in California is relatively small, although reports warrant the statement that California is capable of supplying the entire home demand.

(2) The cost of production of California oil is so much higher than that of the French and Italian oils that it competes with difficulty with the imported oils in the American market, even after the latter have paid duty amounting to 50 cents per gallon.

(3) The retail prices of the best grade of oil from the three sources are much the same, but the average prices of the imported oils are much less than that of the California oil, owing to the large amount of lower grade foreign oils that is marketed in this country.

(4) In the examination of olive oils for adulteration, a complete analysis is usually necessary to reveal the real nature of the oil. In cases of gross adulteration the qualitative tests, specific gravity, and index of refraction will often show the nature of the adulterant and the extent to which it is employed.

(5) The adulteration of foreign oils imported into this country is practiced to a much less extent than is popularly supposed. Only 5 of the 61 samples obtained from the customs officers were found to contain other than olive oil, and none of these contained cotton-seed oil.[a] On the other hand, oils bought upon the market, bearing labels indicating a foreign origin, were found to be quite extensively adulterated with cotton-seed oil. It seems, therefore, probable that these adulterated oils bearing foreign labels are labeled and modified after leaving the port of entry, neither the domestic nor the foreign producer being responsible for them. This practice is equally injurious to the interests of the California, French, or Italian manufacturer of pure olive oil and the consumer.

(6) The results of analyses of oils of known purity show that there is a wide range in the various values ordinarily considered of importance in indicating the purity of an oil. This is especially true of the iodin number, the melting point of fatty acids, and the percentage of solid fatty acids. The California olive oils generally have a higher iodin number, a lower melting point of fatty acids, and a lower percentage of solid fatty acids than the French and Italian oils.

(7) All samples containing other than olive oil were sold as pure olive oil, although in one case a careful observation of the label revealed the fact that the oil was an olive oil substitute.

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[a] Two samples recently received from the custom-house have contained cotton-seed oil.

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U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 78.

H. W. WILEY, CHIEF.

THE INFLUENCE OF ENVIRONMENT
UPON THE
COMPOSITION OF THE SUGAR BEET, 1902,

INCLUDING A STUDY OF IRRIGATED SECTIONS.

BY

HARVEY W. WILEY,
CHIEF OF BUREAU,

IN COLLABORATION WITH THE WEATHER BUREAU AND THE AGRICULTURAL
EXPERIMENT STATIONS OF CALIFORNIA, COLORADO, INDIANA,
KENTUCKY, MICHIGAN, NEW MEXICO, NEW YORK,
UTAH, VIRGINIA, AND WISCONSIN.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1903.

LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., June 30, 1903.

Sir: I have the honor to transmit for your approval a manuscript, accompanied by graphic charts, embodying the results of the cooperative work conducted by this Bureau on the study of the effect of environment upon the composition of the sugar beet during the year 1902, and to recommend its publication as Bulletin 78 of the Bureau of Chemistry. This is a continuation of the work of 1900, the results of which were published in Bulletin 64, and of 1901, reported in Bulletin 74.

In this connection I desire to express the appreciation of this Bureau of the collaborative work done by the various experiment stations taking part in the experiment; of the continued cooperation of the Weather Bureau, without which it would be very difficult, if not impossible, to collect the meteorological data; and of the information furnished by the United States Coast and Geodetic Survey and the Naval Observatory. The analytical work done in this Bureau on the beets was performed by Messrs. Houghton and Given; that on the soils by Messrs. Veitch and Trescot; while the mechanical analyses were made by the Bureau of Soils.

Respectfully,

H. W. Wiley, *Chief.*

Hon. James Wilson,
Secretary of Agriculture.

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THE INFLUENCE OF ENVIRONMENT UPON THE COMPOSITION OF THE SUGAR BEET, 1902.

ORGANIZATION OF COLLABORATIVE WORK.

In the third year of the cooperative experiments with the sugar beet two series of experiments were conducted, one with stations where irrigation was practiced and the other with the nonirrigated stations at which the work has been carried on for two years. The consideration of the soils in which the beets were grown, which was begun in a tentative manner in 1901, was continued and a more complete study was made of this phase of the environment, two additional charts being added to show the relation of the more important plant foods and the physical condition of the soil to the yield per acre.

On February 28, 1902, the following letter was sent to the stations at Logan, Utah; Fort Collins, Colo.; Mesilla Park, N. Mex.; Tucson, Ariz.; Laramie, Wyo.; Moscow, Idaho; Berkeley, Cal.; Pullman, Wash., and Corvallis, Oreg., at which irrigation would probably be practiced:

> DEAR SIR: It is desirable to extend the studies of the influence of environment on the constitution of the sugar beet to areas where irrigation is practiced. The scope of these studies and the methods of conducting them are sufficiently outlined in Bulletin 64 of this Bureau, a copy of which is inclosed.
>
> I desire to have your cooperation in these studies and feel very sure that great good will come of them, not only to the beet-sugar industry, but to agriculture in general. The extent of the collaboration is sufficiently shown in Bulletin 64. If you are willing to engage in these collaborative studies, I beg you to inform me at an early date. The necessary seeds for planting and uniform directions for culture and irrigation will be prepared and forwarded as soon as your acceptance is received. I send this letter with the full approbation of Mr. Elwood Mead, in charge of the irrigation work of the Department of Agriculture, who would have joined me in the signature had he not been absent from the city.
>
> Respectfully,
>
> H. W. WILEY, *Chief.*

Of the stations to which this letter was sent, five—namely, those of Utah, Wyoming, California, Colorado, and Idaho—consented to take part in the experiment. Only three reports are given, however, as the Wyoming crop was a failure and the work done at the Idaho station was not comparable with the other experiments, no irrigation being practiced. The request reached the Arizona station too late in the season for acceptance.

To the stations which had previously cooperated in the work, namely, those of North Carolina, Iowa, Michigan, Virginia, Indiana, Wisconsin, Kentucky, and the two New York stations, the following letter was sent:

MARCH 7, 1902.

DEAR SIR: The good results which have attended the collaborative work in the study of the effect of environment upon sugar beets, and the high commendations thereof which have been received from many practical sources, lead me to believe that a continuance of this work would be highly beneficial. The Weather Bureau has promised to continue its collaboration, and I desire to ask you to undertake again work similar to that which you have done in this direction the past two years. Seeds of high grade and with a germinating power of high quality, as tested in this Department, will be sent to you, as heretofore, for experimental work. The character of the experiments is sufficiently outlined in Bulletin 64, a copy of which I send inclosed.

Hoping that I may have a favorable reply, I am,

Respectfully, H. W. WILEY, *Chief.*

The North Carolina station, which had withdrawn from the work in 1901, was again unable to cooperate, and the work was also temporarily discontinued at Ames, Iowa, on account of a change in the station force.

The following letter containing complete instructions as to the conduct of the experiment, the sampling of the soil, records to be kept, etc., together with an additional note for the stations practicing irrigation, was sent to all cooperating stations at the opening of the season:

APRIL 15, 1902.

DEAR SIR: For the collaborative work in the study of the influence of environment on the composition of the sugar beet, for the present year, I have decided to use seed No. 8238, and a package of this number has been sent to you for such collaborative work.

The area planted need not exceed an eighth of an acre, unless you desire a larger area or a number of plats. This matter is left entirely to your own judgment, and the residue of the seed you can dispose of as you like. I suggest, however, that the special plat be seeded very heavily so as to be sure of a good stand, and that enough of the seed be reserved for replanting in case the first planting should not germinate.

The soil some time before sowing, preferably the previous autumn, should be plowed to the usual depth of 8 or 9 inches and subsoiled to at least 6 inches more, making a seed bed of at least 15 inches in depth. If the character of the soil warrants it, a deeper plowing, even to 10 or 11 inches, and a subsoiling of 6 inches additional, will be advisable.

The surface of the soil should be reduced to a fine tilth and well harrowed and stirred immediately before planting so as to stop all growth of weeds which may have started. The rows should be 18 inches apart and the seed planted at the rate of about 25 pounds per acre, so as to be sure of a good stand. If the soil be moist the seed should be covered to a depth of from one-half to 1 inch. If the weather be dry, slightly deeper planting may be advisable.

As soon as the plants are vigorously growing, they should be "bunched" by a hoe 6 inches in width, leaving the length of 3 inches of beets in each hill. When the beets have a vigorous growth and begin to form the fourth leaf, they should be thinned to about one plant in each 9 inches. Where vacancies occur in a row, transplant carefully so as to have the number of plants indicated.

Ordinary surface cultivation is all that is required, being careful not to cover up the beets at the first cultivation.

In connection with this study, I desire to make careful chemical and physical analyses of the soils of the plats used for the growing of the beets. I therefore ask that you take a representative sample of the soil and subsoil of the plat on which you grow the No. 8238 seed during the present year. After getting a representative sample reduce it in size by quartering or otherwise so as to secure a representative subsample weighing not more than 4 pounds, and send it, under the inclosed frank, by mail to my address.

In sending the sample of soil in accordance with the above instructions, do not forget to send a history of the plat so far as known. Complete cultural and meteorological data, in collaboration with the Weather Bureau, should be kept and forwarded with the samples. Franks for forwarding the samples and full instructions for sampling and harvesting will be sent later. It is earnestly requested that frequent analyses be made at the station, so that the results of those analyses can be compared with those which are made of the beets sent here.

Any questions in regard to further details will be promptly answered.

Respectfully,

H. W. Wiley, *Chief.*

The note to the stations using irrigation was as follows:

IRRIGATED PLATS.

If irrigation is practiced it is desirable that a record be kept of the number of times water is applied and the amount used each time.

In regard to the details of irrigation I can not offer any suggestions, since I have had no experience whatever with irrigated plats. It appears to me that the important thing is to secure the regular and uninterrupted growth of the beets until maturity or near maturity, so that they can ripen without any danger of undergoing a second growth.

In these experiments the temperature record is of more than ordinary importance, since there is some ground for believing that a beet of excellent sugar content can be produced under irrigation where the mean temperature is much higher than is favorable to the production of a good beet in regions under ordinary cultivation.

Since this is the first collaborative experiment in this country undertaken in irrigated areas, suggestions for the second year's work are respectfully solicited.

The seed used in this experiment, referred to as S. P. I. No. 8238, was the Kleinwanzlebener Nachzucht, produced by H. Bennecke & Son, at Athensleben bei Löderburg, Germany, and transmitted to this Department through Ernst Anders, of Magdeburg, for use in this experiment. Mr. Anders, in transmitting the seed, said:

The high germinating power is a very important thing, guaranteeing a quick and equal start, regular growth, and finally a high tonnage. Please take in consideration that the sugar produced per acre will be increased to a very high amount by this fact, notwithstanding the percentage of sugar in the beets may be a little smaller in comparison with other high-grade beet seed giving a smaller tonnage. * * *

Furthermore, the power to germinate very quickly is very important in cases of destruction of the small plants by insects, or if the preparation of the fields is not finished in the proper time, as in either case a late drilling becomes necessary.

The germinating tests of the seed made in the laboratory of Seed and Plant Introduction, Bureau of Plant Industry, of this Department, gave a germination of 80 per cent of seed balls, 196.5 sprouts from 100 seed balls, with 106,000 sprouts from 1,000 grams of seed, thus fully justifying the remarks of Mr. Anders concerning its vitality.

When the time for harvesting approached the following letter was addressed to the cooperating stations:

SEPTEMBER 9, 1902.

DEAR SIR: We are about to begin our analytical work on samples of sugar beets grown from the special seed No. 8238. This is the seed that I requested you to plant for the collaborative work in a study of the influence of environment on the composition of the beet. If practicable I would like you to follow the plan outlined below in obtaining samples of beets.

Harvest every beet in 50 feet of an inside row; wash, dry, and weigh the beets after removing the leaves, but not the crown or neck. Report the estimated yield per acre based upon the weight of the beets (not topped) from 50 feet of row. Forward to this Bureau by express, charges collect, about twenty-five average beets (not topped) from those harvested. Please weigh the beets before shipment, that we may calculate the loss in weight during transit to Washington. We shall use your estimated tonnage and the percentage of loss in topping in estimating the net yield per acre.

Please repeat this work every week until the end of the season. An identification slip giving date, name of station, and the weights and data requested above should be inclosed with each sample. With the first sample forward agricultural data.

Begin sampling on receipt of this letter, not awaiting full maturity of the beets. From time to time I will report the results of the analyses of the samples to you.

Trusting that it will be convenient for you to continue the cooperative work as outlined above, I am,

Respectfully,

H. W. WILEY, *Chief.*

To distant stations cooperating in the work the following additional suggestions were made:

Since your experimental fields are so far from our laboratory, I would be very glad if you could arrange to have these analyses made at your station. If you can not do this, it will be necessary to subsample the beets harvested, report the agricultural data, and pack a carefully weighed quantity of untopped beets in a box and ship them to this laboratory, reporting the weight shipped. In this event about twenty beets should be sent.

I trust that it will not be necessary to forward these samples, as the results will be much more satisfactory if obtained with the fresh material at your laboratory.

These data should be obtained from the beets grown on both the irrigated and non-irrigated plats.

EXPERIMENTS CONDUCTED IN HUMID REGIONS.

POTOMAC FLATS, WASHINGTON, D. C.

Two series of experiments were carried on at the Department farm, Potomac flats. Each consisted of four plats, with dates of planting varying from April 18 to May 30, and in one series irrigation was practiced. The cultural data, except as to irrigation, were the same for both series of plats and are given as follows:

Plat 1.—Planted April 18; up April 22; thinned to two in a hill April 28, 29; thinned to one in a hill on May 14. The plat was cultivated with a wheel hoe on April 25, May 2, 8, 16, 26, June 12, and June 25, when the crop was laid by.

Plat 2.—Planted May 2; up May 8; thinned to two in a hill May 16; thinned to one in a hill May 24. The plat was cultivated on May 16, 26, June 12, 25, 30, and July 2, when the crop was laid by.

Plat 3.—Planted May 16; up May 20; thinned to one in a hill June 6. The plat was cultivated on May 26, June 6, 12, 18, 25, 30, July 5, and July 11, when the crop was laid by.

Plat 4.—Planted May 30; up June 10 (up where irrigated June 5); thinned to one in a hill June 24. The plat was cultivated on June 12, 18, 25, 30, July 5, and July 11, when the crop was laid by.

Each plat or planting consisted of 12 rows, each 25 rods long. The seeds were run in with a seed drill having the dropping attachment set to 9 inches, the rows being 18 inches apart. The plants were thinned as soon as the seedlings were large enough to handle.

The following record shows the amount of moisture received by the irrigated plats from both rainfall and irrigation. By leaving out of consideration the dates followed by the expression (I), which indicates moisture received by irrigation, the amount of water received by the nonirrigated plats can be determined.

1902.	Inches.	
April 29	0.21	
30	.11	
Total		0.32
May 3	0.36	
	0.16	
	.06	
	.98	
	1.18	
	.65	
	1.93	
	.35	
Total		5.67
June 2 (I)	0.50	
5 (I)	.50	
7	.57	
11	.15	
12 (I)	.50	
16	.93	
19 (I)	.50	
19	.21	
21	1.22	
25	.05	
26	1.60	
30	.37	
Total		7.10

1902.		Inches.
July 3 (I)	0.50	
5 (I)	.25	
9	.45	
14 (I)	.60	
16 (I)	[a] .45	
18	1.31	
19	Trace.	
20	.19	
21	.22	
24	.16	
30	.75	
31	.70	
Total		5.58
Aug. 4	0.25	
6	.94	
10	1.02	
23	.20	
26	.38	
27	.04	
Total		2.83
Sept. 3	1.03	
9	1.65	
Total		2.68
General total		24.18

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[a] Applied to plats Nos. 3 and 4 only.

The analytical and field data, determined for the different plats grown under varying conditions, are shown in the following table:

Agricultural and analytical data on beets grown on the Potomac flats, District of Columbia, showing averages for different dates of planting.

UNIRRIGATED.

Plat No.	Date planted.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
	1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
1	Apr. 18	18.6	27.9	9.1	8.3	72.3
2	[a]May 2	22.9	26.1	9.2	8.4	72.4
3	May 16	10.6	19.6	9.3	8.5	72.4
4	May 30	9.9	14.4	9.3	8.6	72.1
Average	....	15.5	22	9.2	8.5	72.3

IRRIGATED.

Plat No.	Date planted.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1	Apr. 18	23.5	18.3	7.7	7.1	67.9
2	May 2	25.4	15.8	8.0	7.4	69.5
3	May 16	12.5	10.8	8.7	8.0	71
4	May 30	12.9	10.8	8.9	8.1	70.5
Average	....	18.6	13.9	8.3	7.7	69.7

[a] The data for May 2, unirrigated, are platted.

The climatic conditions under which this crop was grown are shown by the following data:

Meteorological data for Washington, D. C.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	° *F.*	*Inches.*	*Hours.*	*Hours.*			
May	65.4	3.35	290.0	443.8	65	13	4
June	71.8	3.70	306.3	445.9	69	14	5
July	77	2.54	304.6	453	67	11	3
Average and total	71.4	9.59	....	....	67	38	12
August	72.6	1.85	321	423.2	76	11	4
September	66.8	5.30	218.4	373.4	58	13	3
October	57.6	6.76	229.1	346	66	18	6
Average and total	65.7	13.91	....	....	67	42	13
General average and sum total	68.6	23.50	....	....	67	80	25

The rather contradictory data relative to the yields of the unirrigated and irrigated plats call for a word of explanation. The irrigated plats were located at the west end of the field, the rows of beets running east and west. Unfortunately the stand of beets upon this end was not so good as on the central and western portions of the area planted with beets. There were some places in the rows where beets failed to grow, and thus in thinning fewer beets were left in a given length of row on the irrigated than on the unirrigated portion. A single illustration taken at random will illustrate this. On September 16, 50 feet harvested from each of the four plats planted on April 18, May 2, May 16, and May 30 gave the following number of beets respectively: 59, 49, 60, and 54—total, 222. On the same day 50 feet in length from each of the four rows of irrigated beets yielded the following number of beets: 26, 26, 33, and 23—a total of 108, or less than one-half as many as were yielded by the unirrigated plats. This is of course an extreme case, but the same general proportions as to the number of beets harvested on the two plats obtained throughout the season. This condition explains the apparent contradiction in the yields of the two plats since the unirrigated portions, although the beets weighed less, yielded a larger tonnage than the irrigated plats.

By reason of the larger size of the beets on the irrigated area it is not surprising that they were inferior both in sugar content and in purity to those taken from the unirrigated areas. It does not appear that the practice of irrigation had any effect upon the crop. In fact, it was a most unfavorable season to show any good effect of irrigation, since the rainfall, with the one exception of the month of August, was entirely sufficient in quantity and was advantageously distributed. The excess of rainfall in September and October of course had a tendency to continue the growth and retard the period of maturity. Nevertheless, the beets produced were decidedly superior to those of preceding years. This is probably explained by the fact that the mean temperature of the six growing months in 1900 was 71.7°; in 1901, 69°; and in 1902, 68.6°—a steady though slight decrease.

THE INDIANA STATION.

Unfortunately there are no data to be presented for the Indiana station as the crop was a complete failure. Director Huston comments on this fact under date of January 16, 1903, as follows:

> The seed which was received from you was planted upon ground carefully prepared and germinated satisfactorily. On May 20 the beets were up and showing a good stand. On June 10 there were practically no live beets in the field, and Mr. Jones at once had a portion of the field replanted with the reserved seed. This planting also germinated reasonably well, but the enormous rainfall in June destroyed the young beets, so that there was practically nothing left upon the field. We later put soy beans on the field and they failed to grow. I am rather at a loss to understand what was the cause of these failures, as the field had been under two different crops since beets had been raised upon it. I am under the impression that the first planting was injured by the first cultivation, but as the man who attended to it is not here I am unable to be sure that this was the real cause. Last year a crop of soy beans was plowed under on this land and I thought it was in fine condition and had arranged to irrigate a portion of the field and had all the pipes in place for this purpose. Since the failure of the crops on this piece of ground this spring it has been kept in fallow, and I will have it thoroughly prepared this fall and think it ought to be in first-class condition for the beet work next year.

The meteorological data for this season at Lafayette and at Indianapolis, 59 miles to the southeast, are given in the following tables:

Meteorological data for Lafayette, Ind.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	°*F.*	*Inches.*	*Hours.*	*Hours.*			
May	65.3	3.64				7	16
June	68.4	11.37				5	15
July	74.8	6.56				11	8
Average and total	69.5	21.57				23	39
August	70.1	1.40				12	13
September	63.3	3.72				10	15
October	56.1	3.27				10	7
Average and total	63.2	8.39				32	35
General average and sum total	66.4	29.96				55	74

Meteorological data for Indianapolis, Ind.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	°*F.*	*Inches.*	*Hours.*	*Hours.*			
May	66.0	3.66	300.8	446.7	67.0	4	3
June	69.5	7.52	282.6	449.0	63.0	9	8
July	75.8	3.67	330.9	455.2	73.0	10	3
Average and total	70.4	14.85			67.7	23	14
August	71.6	2.09	262.2	425.2	62.0	10	3
September	64.4	5.33	190.3	373.6	51.0	8	9
October	57.4	2.36	214.1	344.9	62.0	9	5
Average and total	64.5	9.78			58.3	27	17
General average and sum total	67.5	24.63			63.0	50	31

THE KENTUCKY STATION.

The same plat that had been used for the beet experiments in 1900 and 1901 was again assigned for that work in 1902. The land was plowed on April 16, replowed and subsoiled 20 inches deep on April 22. The soil was put in perfect tilth with a smoothing harrow and roller. The seed were planted on the same day about 1 inch deep in rows 18 inches apart, a hand drill being used. The plants were up on April 30, and were cultivated as follows: May 9, hoed; May 14, hoed and plowed; May 21, thinned to 6 inches, hoed and plowed; May 28, thinned to 9 inches and cultivated. At the latter date there was almost a perfect stand, and plants were transplanted to fill the few vacancies that occurred. Throughout the growing season the beets were cultivated once every two weeks.

The early growth of the crop was very vigorous, and the season until September 1, was favorable. During September and October the beets grew very little, but a considerable growth took place in November.

The analytical data on the samples taken as determined both at Washington and at Lexington are given in the following tables:

Agricultural and analytical data on beets grown at Lexington, Ky.

Date received.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 18	8.0	10.0	9.3	8.6	74.6
October 9	8.6	9.0	7.4	6.8	68.5
October 21	8.0	8.0	7.5	6.9	70.7
October 25	7.2	8.4	7.6	7.0	69.7
Average	8.0	8.9	8.0	7.3	70.9

Sugar beets grown and analyzed at Lexington, Ky.

Date of sampling.	Number of beets taken.	Average weight after topping.	Sugar in the juice.	Sugar in the beet.[a]	Purity coefficient.
1902.		*Ounces.*	*Per cent.*	*Per cent.*	
June 25	7	4.7	8.15	7.50	65.7
August 6	12	12.4	9.53	8.77	73.8
August 19	9	13.7	9.30	8.56	72.3
October 4	4	9.2	7.15	6.58	66.0
October 13	5	8.8	4.75	4.37	57.2
October 21	6	10.0	6.92	6.37	66.1
December 4	6	17.1	8.77	8.07	67.0
December 8	5	14.6	8.38	7.71	67.6
Average	....	11.3	7.87	7.24	67.0

[a] Calculated at Washington, D. C.

The meteorological conditions existing during the growing season of 1902 at Lexington are shown in the following table:

Meteorological data for Lexington, Ky.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	° *F.*	*Inches.*	*Hours.*	*Hours.*			
May	68.2	2.43	364.5	441.7	83.0	18	0
June	71.2	5.19	359.3	443.1	81.0	13	4
July	76.8	2.33	361.4	450.1	80.0	14	0
Average and total	72.1	9.95	....	....	81.1	45	4
August	73.8	1.89	330.4	422.1	78.0	13	2
September	67.0	2.60	245.1	373.0	66.0	10	7
October	59.0	2.11	241.2	347.3	69.0	15	9
Average and total	66.6	6.60	....	....	71.0	38	18
General average and sum total	69.3	16.55	....	....	76.1	83	22

The following table, compiled at the Kentucky station, shows the averages in temperature and precipitation for fifteen years prior to 1903, and also the accumulated deficiency of precipitation from January 1, 1902:

Averages for fifteen years, and accumulated rainfall deficiency.

Meteorological data.	May.	June.	July.	Aug.	Sept.	Oct.
Temperature....degrees F..	64	74	76	74	69	57
Precipitation....inches..	3.71	4.31	4.06	3.95	2.59	2.11
Accumulated rainfall deficiency from Jan. 1, 1902–Jan. 1, 1903.[a]....inches..	7.36	6.39	8.12	10.18	10.17	10.17

[a] The accumulated deficiency of precipitation for the preceding year, 1901, was 13.28 inches.

Under date of February 11, 1903, Mr. A. M. Peter, chemist of the station, forwarded some additional data obtained on beets from the experimental plat which were harvested in the second week of November and stored in a cellar until January 31, 1903, when they were sampled and the following analyses made:

Data determined on beets stored for two months at Lexington, Ky.

Station number.	Number of beets.	Weight of beet.		Sugar in juice.	Purity.
		Topped.	Trimmed.		
		Ounces.	*Ounces.*	*Per cent.*	
10892	1	16.2	13.2	7.4	72.6
10893	1	15.7	12.0	7.2	59.5
10894	1	15.0	12.5	9.95	71.8
10895	1	16.8	11.8	9.1	72.2
10896	1	13.0	10.5	8.1	65.3
10897	2	10.1	7.9	9.55	71.8
Average	....	14.5	11.3	8.6	68.9

The results of the experiment at the Lexington station are particularly interesting in that they have again robbed the Washington station of its original position at the foot of the ladder in the production of beets of a low sugar content. The precipitation at Lexington, while below the average, appears to have been sufficient for the needs of the crop. The month of August, however, as at Washington, was somewhat dry. The average temperature for the six growing months was 69.3° or 0.7° higher than at Washington.

THE MICHIGAN STATION.

Under date of May 16, 1902, Director Smith reported that the beets were up and large enough to be cultivated within a week. The season was wet and cold, frosty nights occurring especially during May. The crop harvested was of very high quality, however, as is shown by the following table:

Agricultural and analytical data on beets grown at Agricultural College, Mich.

Date received.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 20	10.9	....	15.1	13.9	82.1
September 27	10.9	....	15.0	13.8	86.7
October 7	10.2	12.8	14.4	13.2	86.2
October 14	10.2	12.0	14.4	13.2	86.7
October 21	11.5	14.5	14.8	13.6	87.6
October 28	12.5	....	15.2	13.9	89.4
November 4	8.3	10.5	15.0	13.8	89.3
Average	10.6	12.5	14.8	13.5	86.9

The meteorological data for Agricultural College and for Detroit, 76 miles to the southeast of the college station, are given in the following tables, the sunshine record not being available for any point nearer to Agricultural College than Detroit:

Meteorological data for Agricultural College, Mich.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	°*F.*	*Inches.*	*Hours.*	*Hours.*			
May	58.4	4.92	....	....	....	6	11
June	61.8	7.28	....	....	....	9	19
July	70.6	7.13	....	....	....	....	....
Average and total	63.6	19.33	....	....	....	15	30
August	64.2	.68	....	....	....	11	7
September	58.7	5.88	....	....	....	11	17
October	49.6	1.53	....	....	....	18	13
Average and total	57.5	8.09	....	....	....	40	37
General average and sum total	60.5	27.42	....	....	....	55	67

Meteorological data for Detroit, Mich.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	°*F.*	*Inches.*	*Hours.*	*Hours.*			
May	58.4	3.52	298.7	451.9	66	12	6
June	63.9	6.97	235.6	456.2	52	5	9
July	72.8	7.55	291.9	461.8	63	13	7
Average and total	65.0	18.04	....	....	60	30	22
August	68.0	.60	285.8	429.4	67	15	5
September	62.2	6.50	186.4	374.5	50	10	12
October	52.4	1.54	177.5	342.5	52	10	11
Average and total	60.9	8.64	....	....	56	35	28
General average and sum total	63.0	26.68	....	....	58	65	50

The precipitation for the early growing months of May, June, and July was excessive, especially for the two months last named. During the second period of three months the August precipitation was deficient and the September rainfall was excessive, while October was somewhat dry and favorable to securing the maturity of the plants. It is evident that had two-thirds of the September rain fallen in August there would have been a larger yield of beets and perhaps of a better quality. Both the percentage of sugar in the beet and the purity of the juice, however, are very satisfactory.

THE NEW YORK STATION AT GENEVA.

The following cultural data are reported by the Geneva station:

The seed was sown on May 30, 1902. The ground was plowed and subsoiled in the spring to the depth of 14 to 16 inches. The fertilizer intended for the work was mixed and weighed out, but the fire of May 7 destroyed it and before a new supply could be obtained it was too late, and therefore no fertilizer was used.

On June 4 the seed began to vegetate and growth went on rapidly. A very uniform stand was obtained without reseeding. On June 14 the beets were cultivated by hand, and three subsequent cultivations with a one-horse cultivator were given. Thinning was begun on June 27 and finished on June 30. The beets were thinned to approximately 8 inches in the rows, the rows being 20 inches apart. The beets were practically free from leaf spot and grew with vigor throughout the season. The data on the beets determined at the New York station are as follows, each sample representing 50 beets.

Agricultural and analytical data determined at Geneva. N. Y.

Date harvested.	Average weight.	Yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
October 14	13.8		17.1	14.9	91.9
November 13	17.5	16.06	17.1	14.3	87.4
Average	15.7	16.06	17.1	14.6	89.7

The data determined at Washington, D. C., on the beets forwarded from Geneva are shown in the following table:

Agricultural and analytical data on beets grown at Geneva, N. Y.

Date received.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 22	10.6		15.1	13.9	83.9
September 30	13.1		15.7	14.4	84.4
October 9	14.4		14.1	12.9	83.9
October 14	17.4		14.1	12.9	83.4
October 21	14.7		15.1	13.9	86.3
October 29	14.7		15.3	14.1	85.5
November 4	12.5		15.9	14.6	85.0
November 11	17.6		15.6	14.4	84.3
November 18	13.7		15.3	14.1	84.1
Average	14.3	[a] 16.1	15.1	13.9	84.5

[a] Estimate made at Geneva.

Unfortunately only very scanty meteorological records are procurable for this locality, the temperature and precipitation record kept at the voluntary station at Lyons, 13 miles north of Geneva, furnishing the only data available except rainfall reported from Geneva in the following paragraph.

Meteorological data for Lyons, N. Y.

Month.	Mean temperature.	Precipitation.
1902.	*°F.*	*Inches.*
May	58.8	3.03
June	64.0	3.39
July	72.4	6.77
Average and total	65.1	13.19
August	67.8	2.22
September	64.4	2.57
October	51.0	2.79
Average and total	61.1	7.58
General average and sum total	63.1	20.77

The following comments on the season are made in the station report on the experiment:

> The season was an unusual one in some respects. Periods of excessive moisture were followed by drought, making it difficult to give the crop frequent cultivation. The rainfall as observed at Geneva for the growing season was as follows:

	Inches.		Inches.
June	4.33	October	2.32
July	5.25		
August	2.41	Total	17.19
September	2.88		

> In June the longest period without rain was three days. From July 9 to 15 was dry. In August the longest period with no rain was five days, from August 12 to 17. In September there was but 0.75 inch from the 1st to the 22d. The coldest October and the warmest November were experienced that have been known in this latitude for twenty years.

The analytical data determined at the Geneva station and those obtained at Washington vary somewhat, the Geneva figures for purity and sugar in the beet being higher. Only two samples, however, were examined at Geneva, and those were of the perfectly matured beets, the samples having been taken on October 14 and November 13, while the samples received at Washington covered a range of harvesting from the last of September to the middle of November. The difference in the percentages of sugar in the beets as obtained at the two stations is only 0.7 per cent, while the difference in the purity coefficients is 5.2 per cent. Following the usual rule, the data obtained in this laboratory have been platted in preference to those of the collaborating station. This is not done because of any assumed greater degree of accuracy, but because the analyses made here are all performed under standard and uniform conditions. In point of fact there may possibly be some slight deterioration in the samples sent to Washington, especially those coming a long distance. This deterioration can never be very great, however, if the directions for packing and shipping are followed carefully.

THE NEW YORK STATION AT ITHACA.

The following report on the beet work as conducted at the Ithaca station was made by Mr. J. L. Stone, assistant in agriculture, under date of December 4, 1902:

> The sugar-beet seed No. 8238 of the Department of Agriculture was planted May 13, 1902, at the extreme southwest corner of the permanent series of plats. * * * The land was plowed early and harrowed at intervals till the time of planting. Seed was sown with a hand drill, the distance between the rows being 22 inches. The dates of cultivation seem not to have been recorded, but the crop was well cared for, no weeds being observed when the plats came into the writer's care in September. The plants were thinned to a stand on June 19. When harvesting began on September 18 the beets were immature, and the soil had been very wet most of the time since early June. The last days of September were bright and sunny. October was nearly normal as regards precipitation, but the soil was still quite wet from earlier rains. November, up to the 25th of the month, was unusually warm, with little rainfall. Second growth had taken place to a considerable extent. * * * The following are the dates of harvesting and data relating to the same:

Agricultural data determined at Ithaca.

Date of harvest.	Number of beets in 50 feet.	Total weight with crowns.	Weight of 25 beets.	Dockage.	Estimated yield per acre (trimmed).
1902.		*Pounds.*	*Pounds.*	*Per cent.*	*Tons.*
September 18	67	68.0	25.0	10.4	14.47
September 25	66	70.0	26.0	12.5	14.55
October 2	62	82.0	35.0	8.5	17.83
October 9	64	82.0	33.0	8.5	18.24
October 17	71	87.0	34.5	10.0	18.61
October 25	60	91.5	37.0	9.3	19.72
November 1	63	85.5	33.5	10.5	18.18
November 10	58	79.5	35.0	9.0	17.19
November 17	63	94.5	37.0	8.7	20.75
November 25	60	102.5	45.0	14.0	20.94
Average	63	84.3	34.1	10.1	18.05

> Evidently the per cent of dockage was materially increased at the last date owing to the second growth and the consequent increase in the weight of the crown. However, it is evident that at different dates of harvest the manner of trimming the beets was not quite uniform or the per cent of dockage would not vary so irregularly.

The following tables show the data determined at Washington on the beets shipped from the Ithaca station, and also the climatic conditions under which they were grown:

Agricultural and analytical data on beets grown at Ithaca, N. Y.

Date received.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 22	13.1	10.9	13.1	12.1	81.9
September 30	13.8	10.9	12.9	11.9	82.7
October 7	17.3	14.1	11.7	10.8	79.1
October 14	16.6	20.1	12.1	13.9	79.6
October 21	17.9	20.5	13.2	11.1	81.5
October 29	19.8	21.4	14.0	12.9	83.8
November 7	16.6	20.0	14.2	13.1	85.0
November 14	17.6	19.2	14.3	13.1	85.6
November 22	16.0	19.9	14.4	13.2	82.7
November 29	21.6	23.0	13.6	12.6	76.8
Average	17.03	18.0	13.3	12.5	81.87

Meteorological data for Ithaca, N. Y.

Month.	Mean temperature.	Precipitation.	Clear days.	Cloudy days.
1902.	° *F.*	*Inches.*		
May	54.5	2.05	8	10
June	62.2	5.39	4	5
July	69.8	6.69	6	10
Average and total	62.2	14.13	18	25
August	65.6	3.13	9	6
September	61.1	2.70	11	8
October	49.2	3.36	3	15
Average and total	58.6	9.19	23	29
General average and sum total	60.4	23.32	41	54

The agricultural data from the Ithaca station are very full and satisfactory, but no chemical analyses of the samples were made at the station. The results of our analyses show, as in previous years, that the beets grown at the Ithaca station are slightly inferior to those produced at the Geneva station, although the difference of location of the two stations is not very great. A comparison of the climatic conditions existing at the two stations is not directly possible, because practically no data are given for Geneva itself. In comparing the meteorological data given for Lyons, 13 miles from Geneva, with the Ithaca report, we find that the temperature at Ithaca is lower than at Lyons, which would be contrary to the general rule that the lower temperature favors the production of a richer beet.

THE VIRGINIA STATION.

Details in regard to the sugar-beet experiment at the Blacksburg station were reported by Mr. Alwood, under date of September 13, 1902, as follows:

The land chosen for the plat this year is a fairly strong clay loam which had been farmed in corn the two previous years and heavily cropped, using some phosphate;

how much I can not say positively. This land came to the garden department last fall. We covered it very heavily with manure and plowed it in the late fall, and it was left rough all winter. This spring before planting we harrowed the land severa times and then on April 30 replowed and subsoiled it down to 18 inches. The land was put in fine tilth before planting. There was so much manure in the soil that the soil samples were not taken at that time. * * * On May 1 the plat was sown with seed No. 8238, using a garden drill, and placing the rows about 18 inches apart. The seed was sown thickly in the row. The season turned exceedingly dry from May 1 and has so continued up to this date, yet the fine condition of the land has enabled us to grow a very fair crop.

Vegetation occurred in one week, and for several weeks the land was cultivated with a hand tool, giving a shallow cultivation once each week, and the beets were thinned as directed in the instructions. By June 30 the growth of tops was so luxuriant that the ground was practically hidden from view. On September 1 the outer leaves began to fall down and rest on the surface of the soil. On this date 50 feet of row was sampled, with the following results:

Number of beets in 50 feet of row	72
Weight of beets, leaves removed only ..... pounds	76
Weight of 25 beets not topped ..... do	31⅝

Under date of September 19 the following additional report was made:

I sent you yesterday the second sample of beets, and the data concerning the same are as follows:

Fifty feet of row in this case dug only 57 beets. It happened that this section of row had a number of vacant spots. I have already mentioned that the drought was very severe here at the time of planting. This number of beets weighed, after taking off the leaves, 60 pounds. The weight of the 25 beets sent to you was 27¾ pounds.

There was a very heavy frost here last Sunday morning, and a somewhat slighter frost occurred Monday morning [September 14 and 15]. The result is that the tops of the beets are now much fallen over, though they are not blackened nor killed, but I think the crop is practically at full maturity, or at least will be next week.

The data obtained on the samples of these beets shipped to Washington from the Blacksburg station are given in the following table:

Agricultural and analytical data on beets grown at Blacksburg, Va.

Date received.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 16	16.0	....	12.0	11.0	71.4
September 20	13.8	....	13.3	12.2	78.7
September 27	16.6	....	12.8	11.8	73.6
October 9	15.0	....	12.9	11.9	73.7
Total	15.4	[a] 16.7	12.8	11.7	74.4

[a] Approximate estimate made on data given in reports of September 1 and 19.

The meteorological data reported for Blacksburg are as follows:

Meteorological data for Blacksburg, Va.

Month.	Mean temperature.	Precipitation.	Clear days.	Cloudy days.
1902.	°F.	*Inches.*		
May	63.9	1.85	20	2
June	68.4	2.90	10	8
July	72.5	4.09	10	6
Average and total	68.3	8.84	40	16
August	71.3	1.06	9	8
September	63.2	2.22	13	9
October	55.1	3.06	12	13
Average and total	63.2	6.34	34	30
General average and sum total	65.8	15.18	74	46

Although Blacksburg is considerably south of both Washington and Lexington, the mean temperature for the six months of the growing season is 2.8° less than at Washington and 3.5° lower than at Lexington. The low latitude of course produces a shorter length of day for the growing season, so that the hours of sunlight at Blacksburg are fewer than at Washington or Lexington. Nevertheless the low temperature occasioned by the great altitude is reasonably favorable to the production of beets with a high content of sugar. The purity of the juices, however, is far below the standard for beets of a richness of almost 12 per cent. The yield per acre is fairly satisfactory.

THE WISCONSIN STATION.

A detailed and very interesting report on the sugar-beet experiment, as conducted at Madison, was made by Mr. F. W. Woll, station chemist, and Mr. R. A. Moore, agriculturist. The report is given practically in full, as follows:

The land on the university farm devoted to sugar beets during the past season was about two-fifths of an acre in area. The soil is a clay loam with a heavy clay subsoil and has a decided tendency to bake after rains, a thick, solid crust being then formed on the surface. During the frequent heavy rains in June and July the soil became quite hard and compact, and the effect of this was seen in the shape of the beets harvested, many of them being forked and abnormally thick and short.

The field, which lies in the western portion of the experimental plat grounds, was divided into 7 plats of one-twentieth of an acre each. Cereal and rape crops were grown on plats Nos. 1 to 5 during the two previous seasons, while the two plats farthest north (Nos. 6 and 7) were in clover during these years. The field was in a high state of fertility. A medium application of barnyard manure was put on plats Nos. 1 to 5 in the fall of 1901 previous to plowing, while plats Nos. 6 and 7 were plowed about 6 inches deep in the spring of 1902. In addition to barnyard manure three of the plats received the following artificial fertilizers: Plat No. 4, 50 pounds of Armour's Sugar-Beet Grower, 29 pounds of nitrate of soda, and 20 pounds of sulphate of potash. The commercial fertilizers were harrowed in directly before planting, except in case of the nitrate, of which one-half was applied at planting time and the remainder when the beets were thinned.

The beet field was disked and harrowed on April 15, and again on April 24, when the seed bed was carefully prepared and the seed planted in rows 18 inches apart, at the rate of 20 pounds to the acre. The field was harrowed lightly after the seed was put in. It was cultivated with a hand cultivator on May 8, 19, 22, and June 3. The beets were hand-hoed on May 22 and June 20, and thinned on May 26, so that the plants stood approximately 9 inches apart in the row. The field did not receive any further treatment after June 20 until harvesting time, except that all weeds were pulled by hand on July 21. The stand of the beets was perfect and the field presented a fine appearance, the beets looking thrifty and strong throughout the growing period.

Owing to the abundance of moisture during the summer months and the cool weather during August and September the beets matured later than is usually the case in this locality. Samples of the beets grown on plat No. 4 were taken every week from September 14 to October 11, as requested by the Bureau of Chemistry, United States Department of Agriculture. The beets in 50 feet of row were dug and weighed before and after being washed, 3 beets of average size being taken in each case for a sample. The results of the weighings and analyses made on the different dates of sampling, as obtained at this station, are shown in the following table:

Agricultural and analytical data determined at Madison, Wis.

Date of sampling.	Average weight.		Estimated yield per acre (washed).	Sugar in juice.	Sugar in beet.	Purity coefficient.
	Beets as dug; topped at crown.	Washed; topped below leaf buds.				
1902.	*Pounds.*	*Pounds.*	*Tons.*	*Per cent.*	*Per cent.*	
September 14	1.5	1.03	30.0	14.4	13.3	78.6
September 20	1.6	1.17	27.3	14.3	13.2	79.3
September 27	1.8	1.58	34.8	14.0	12.9	83.3
October 4	1.8	1.58	33.1	15.7	14.4	81.0
October 11	....	1.26	29.7	13.8	12.7	77.4
Average	1.7	1.32	31.0	14.4	13.3	79.9

Single analyses do not always give a correct indication of the maturity of the beets at the time of sampling, and the results, therefore, should not be interpreted too strictly. But slight improvement occurred in the sugar content or purity of the beets after the first sampling, as far as could be observed.

The following table shows the data obtained at the Bureau of Chemistry for the samples shipped from the Madison station on the dates of sampling given in the preceding table:

Agricultural and analytical data on beets grown at Madison, Wis., as determined at Washington, D. C.

Date received.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 17	17.8	....	13.6	12.5	82.4
September 30	20.2	27.3	14.0	12.9	80.5
October 1	16.3	35.0	14.5	13.3	84.8
October 9	25.9	33.0	13.0	12.0	79.3
October 21	40.6	....	13.9	12.8	83.2
Average	24.2	31.8	13.8	12.7	82.0

The report of the Wisconsin station continues as follows:

Usually beets grown in this locality are ready for harvesting during the first half of the month of September. The beets this year, however, were not harvested until October 11. Those grown on the different plats were of course kept separate, while those grown in the spaces between the plats were thrown together and sampled separately, the data for these beets being given under plat No. 8 in the following table:

Agricultural and analytical data determined at Madison at the time of harvest.

Plat No.	Average weight.	Yield per plat.[a]	Yield per acre.	Sugar per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
	Pounds.	*Pounds.*	*Tons.*	*Pounds.*	*Per cent.*	*Per cent.*	
1	1.34	3,196	32.0	8,371	14.22	13.08	85.2
2	1.42	3,042	30.4	8,637	15.49	14.25	82.6
3	1.27	3,115	31.2	8,811	15.37	14.14	87.3
4	1.26	2,974	29.7	7,489	13.77	12.67	81.0
5	1.44	3,054	30.5	8,333	14.85	13.66	83.1
6	1.42	2,814	28.1	7,789	15.06	13.86	85.1
7[b]	1.46	2,755	27.6	7,529	14.83	13.64	88.4
8	1.12	2,533	29.9	8,647	15.72	14.46	85.0
Total and average	1.34	23,483	29.9	8,213	14.91	13.72	84.5

[a] Each plat about one-twentieth of an acre.
[b] Beets taken from spaces between plats.

The most striking feature of the results presented in the preceding table is the large tonnage. More than 11 tons of washed beets were harvested from an area of only about two-fifths of an acre, which represents a yield of nearly 30 tons per acre. This is an extraordinary tonnage, which has never been equaled in previous trials with sugar beets at this station. The following summary shows the average yields of sugar beets obtained at the university farm during the past twelve years:

Average annual yield of sugar-beet crop for twelve years.

Season.	Yield per acre.	Sugar per acre.	Season.	Yield per acre.	Sugar per acre.
	Tons.	*Pounds.*		*Tons.*	*Pounds.*
1890	19.80	5,913	1900	16.99	3,862
1891	7.34	2,267	1901	10.97	2,623
1892	11.31	3,821	1902	29.94	8,213
1897	9.13	2,503			
1898	18.71	5,312	Average	15.89	4,258
1899	18.83	5,805			

The unusually large tonnage of 1902 is no doubt to be attributed to the following three causes: The abundant supply of moisture during the growing season; the high state of fertility of the soil on which the seed was planted; and the long vegetation period which obtained, the seeds having been planted and the beets thinned nearly one month earlier than had been the practice in previous years. While the beet field was not large there would have been no difficulty in securing a yield of the same proportions for a larger area had there been a sufficient force at the University farm to cultivate it.

The expense of growing an acre of beets under ordinary conditions is generally estimated to be from $25 to $30. Beets of the quality of those grown at the University farm during the past season are paid for at the sugar factory at Menomonee Falls, Wis., at the rate of $4.42 per ton. This would make the product from an acre worth $132.60, a net profit of upward of $100 per acre. The expense of the labor required to keep the field free from weeds was higher this year than in ordinary seasons on account of the difficulty in getting to the field at the right time after the many heavy rainstorms during the early part of the summer. The profit of growing an acre of beets with a yield like that secured during the past season would at all events be very large. The results of investigations conducted at this station in the past have shown that even if the yield of beets should be only one-half of that obtained this year the beet crop would still be a very profitable one for Wisconsin farmers to engage in if they are within a reasonable distance of a beet factory. Estimating the value of the beets and the cost of growing them as above, there would still be a net profit on a crop of half the size of that of 1902 of about $40 per acre of beets grown. It is believed that there are few crops that can be grown in this State which will pay better during a series of years than sugar beets, provided proper attention is given to the crop. As one sugar factory has been in operation in this State for two seasons, and one or two more will in all probability be erected in time for next season's crop the farmers in many localities in our State will soon have an opportunity to engage in the growing of sugar beets for factory purposes. The practical importance of the subject to the farmers of Wisconsin is therefore evident.

In the following table are given the climatic data available for Madison. Mr. Shaw, in commenting on the season, said that the excessive rains of July, followed by almost continuous cloudy weather, while unfavorable for cereal crops, was very favorable to the growth of green forage and root crops, of which large yields were harvested.

Meteorological data for Madison, Wis.

Month.	Mean temperature.	Precipitation.	Clear days.	Cloudy days.
1902.	*°F.*	*Inches.*		
May	59.8	5.16	3	17
June	63.2	4.27	3	18
July	71.3	8.98	4	13
Average and total	64.8	18.41	10	48
August	66.8	.78	11	7
September	58.2	4.18	8	13
October	51.4	1.23	9	8
Average and total	58.8	6.19	28	28
General average and total	61.8	24.60	38	76

The agricultural data obtained from the Wisconsin station are particularly satisfactory in their fullness and detail. The yield per acre, as estimated from the harvested areas, is extremely high. The average weight of the beets is particularly satisfactory, and it is only with beets of this average weight that such high yields can be obtained. In the samples harvested on October 21 and sent to this Bureau the average weight of the beets was phenomenally high, and yet there was no decrease either in the percentage of sugar in the beet or in the purity; in fact, this sample, both in respect of the sugar in the beet and purity, was above the average of the other samples. There is a slight discrepancy between the analytical data obtained at Madison and at this Bureau, in that the figures obtained at Washington for the content of sugar and purity are somewhat lower. The high yield may be attributed to a naturally fertile soil, well prepared, judicious fertilization, timely culture, and a favorable amount and distribution of rainfall. During the first growing period of three months the rainfall was probably excessive, and especially was this true of the month of July. August, as at most of the stations, was a dry month, and September somewhat too wet. October was favorable for ripening and harvesting. The mean temperature was low, thus favoring a high sugar production. The record for this year, upon the whole, must be regarded as extremely satisfactory, and may well be taken as a model for work of this kind.

EXPERIMENTS CONDUCTED IN IRRIGATED SECTIONS.

THE CALIFORNIA STATIONS.

A very complete and interesting report was received from G. W. Shaw, of the California station, in regard to the sugar-beet experiment as conducted at that point. The report is here given practically in full:

The cooperative experiment with sugar beets was conducted under the supervision of the writer at the Pomona substation grounds and on the 10-acre tract at Chino. These localities are but a few miles apart, and their climatic conditions are the same, the only difference in the two localities being in the character of the soil, and in the fact that at Pomona the beets were grown under irrigation and at Chino without irrigation. The soil was prepared by a deep spring plowing, and worked to a fine tilth. The seed was planted on April 2, in drills 20 inches apart, the entire plat being 160 by 75 feet.

Pomona (Irrigated).

The seeds were slow to germinate, and on April 14, the beets on the Pomona tract were irrigated to hasten germination. To secure a uniform stand beets were transplanted to fill vacant spaces in the rows on May 5, and again on May 14, and the plat was thinned from May 6 to 8.

Including the early irrigation mentioned above, the beets received the following-named quantity of water on the dates given:

Irrigation data, Pomona, Cal.

Date of irrigation.	Amount of water remaining on plat.		
1902.	*Gallons.*	*Cubic feet.*	*Acre inches.*
April 14	12,500	1,763	
May 20	5,000	669	
June 17	14,000	1,875	
July 15	8,750	1,172	
July 22	8,575	1,172	
July 29	8,575	1,172	
August 5	8,575	1,172	
Total	65,974	7,823	8.82

The above figures represent the actual amount of water remaining on the plat, as the wastage was estimated as exactly as possible and subtracted from the amount applied. In addition to the 8.82 acre-inches of water received by irrigation, the soil had the benefit of the following natural rainfall during the year:

	Inches.
October, 1901	2.29
November	.67
January, 1902	1.92
February	3.35
March	3.85
April	.25
May	.10
Total by rainfall	12.43
Total by irrigation	8.82
Total water	21.25

Of this total amount, 9.17 inches were applied after planting. It should be stated that the soil is not one retentive of moisture, and except for irrigation would not produce a crop requiring as much moisture as sugar beets, although the season in this locality in other particulars would be considered as favorable for the crop. The analytical and field data on the crop of 1902 are as follows:

Agricultural and analytical data determined at Pomona, Cal.

Date harvested.	Yield per acre.	Sugar in juice.	Sugar in beet.	Purity coefficient.
1902.	*Tons.*	*Per cent.*	*Per cent.*	
September 26		18.7		83.8
October 4		17.8	15.48	85.6
October 11		15.5		91.0
October 18		15.3	14.60	84.5
October 25		15.8	15.00	87.7
November 1		15.1		87.4
November 8		16.8		85.5
Average	5.0	16.4	15.02	86.5

It will be noted that the beets sampled first had the highest percentage of sugar, and it may be that an earlier sampling would have shown even a higher sugar content. During the period of sampling the beets lost about 3 per cent in sugar, but gained that much in purity; hence the actual sugar content remained about the same. There was, however, no decided increase in tonnage. The planting of the beets was decidedly too late for this coast, producing a disastrous effect on the yield, which was but a trifle over 5 tons per acre. The poor quality of the soil also influenced the low yield to a great extent.

The factor showing the relation of sugar in the beet to sugar in the juice, as determined by the hot aqueous method, was 91.6, which my experience leads me to believe to be much nearer the truth for beets in the Western States than the time-honored one of 95 so commonly used and accepted as correct.

CHINO (ALKALI SOIL, NOT IRRIGATED).

The results obtained on the 10-acre alkali tract at Chino are shown in the following table:

Agricultural and analytical data determined at Chino, Cal.

Date harvested.	Estimated yield per acre.	Sugar in juice.	Purity coefficient.
1902.	*Tons.*	*Per cent.*	
September 26	7.40	17.00	87.2
October 4	5.46	17.65	89.6
October 11	13.85	14.70	87.5
October 18	15.71	16.30	89.5
November 1	8.65	13.70	87.7
November 8	9.58	13.10	85.6
December 2	11.71	14.42	88.8
Average	10.34	15.27	88.1

The results on this land are clearly favorable, and show that beets of high quality can be produced upon soil containing at least 5,480 pounds of alkali per acre-foot, and other experiments show that this amount may be exceeded provided the percentage of chlorids and carbonates does not rise too high.

It is noticeable that the beets grown on this tract also fell off materially in sugar content after October 1. This was not due in either case to rainfall nor to irrigation, since from May until November 24 there was no rain, and irrigation was discontinued on August 5 at Pomona. Just why there should be this sudden decrease in sugar is not clear; the fact, however, is quite striking in both cases. The average factor showing the relation between the sugar in the beet and the sugar in the juice for this plat was 93.1.

The meteorological data furnished by the voluntary station near Pomona, considered in connection with those from the Los Angeles station, about 15 miles west of the Pomona and Chino experiment grounds, give a complete record of the climatic conditions for the growing season.

Meteorological data for (near) Pomona, Cal.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	°F.	*Inches.*	*Hours.*	*Hours.*			
May	64.1	0.10				13	2
June	72.5	.23				11	0
July	74.0	.00				6	0
Average and total	70.2	.33				30	2
August	73.8	.00				8	0
September	72.8	.00				11	0
October	62.6	.26				18	4
Average and total	69.7	.26				37	4
General average and sum total	70.0	.59				67	6

Meteorological data for Los Angeles, Cal.

Month.	Mean temperature.	Precipitation.	Sunshine, Actual.	Sunshine, Possible.	Sunshine, Per cent.	Clear days.	Cloudy days.
1902.	°*F.*	*Inches.*	*Hours.*	*Hours.*			
May	60.7	0.03	318.8	432.6	74.0	8	1
June	66.4	Tr.	295.4	431.5	68.0	10	1
July	68.4	Tr.	268.7	439.7	61.0	9	3
Average and total	65.2	.03			67.7	27	5
August	69.1	Tr.	314.0	415.8	76.0	12	0
September	69.2	Tr.	275.2	371.8	74.0	11	1
October	63.2	.40	236.0	350.9	67.0	11	6
Average and total	67.2	.40			72.3	34	7
General average and sum total	66.2	.43			70.0	61	12

The work inaugurated with irrigation stations marks the beginning of the study of a new consideration as affecting the activity of different factors of the environment upon the composition of the crop. In the reports from the California stations just given we find an illustration of what has been well demonstrated regarding the ability of irrigated lands to produce crops of sugar beets phenomenally rich in sugar. At the Pomona station the yield per acre is not satisfactory, although with such rich beets as were there raised the farmer could secure a satisfactory monetary return for the crop produced. The yield was not quite one-half as large as that produced on the Chino plat which was not irrigated. This is evidently a case in which a judicious intensive fertilization would very largely increase the yield without diminishing to any notable extent the percentage of sugar or the purity of the juice, and would, therefore, be highly profitable.

In regard to the meteorological data the principal points to be considered are that, although the mean temperature for the six growing months is higher than at Washington, there were no periods of intense heat; June, July, August, and September had almost an even temperature. At Los Angeles there was also an even temperature, September being the warmest month. The general average for the six months was 3.8° lower than at Pomona. The rainfall at both stations was so small as to be negligible. The high percentage of sunshine is of course to be expected under such meteorological conditions as obtained at the California stations mentioned. It is evident that continuous sunshine, where the temperature does not run too high, is not injurious in any way to the functional activity of the chlorophyll cells. Where sufficient moisture is supplied by irrigation and a proper quantity of suitable plant foods exists, the other conditions combine to make such regions almost ideal for beet culture.

THE COLORADO STATION.

As several sugar-beet experiments were conducted at the Colorado station in 1902, it has been impossible to separate all mention of the other work from that carried on with seed No. 8238 of this Bureau, especially since all the beet plats received the same treatment and were planted at the same time, each plat being, however, clearly distinguished from the others. The following extracts from the general report made by A. H. Danielson, agronomist of the Colorado station, give the details of the conduct of the experiment at Fort Collins:

> The soil this season had been plowed, harrowed, and leveled with a drag until it was in the best possible condition. As a result of the rain and snow storm on April 14 there was plenty of moisture in the soil to insure the germination of the seed. The ground was leveled twice just before drilling in the beet seed, stirring the soil in such a thorough manner as to give the beets an equal chance with the weeds. If anything, the soil was a little too loose and mellow.
>
> One plat of variety No. 8238 was planted on April 17 near the edge of the field where the soil was not in the best possible condition on account of the tramping of the animals in working the soil. However, this condition seemed to affect the three or four outside rows only. To insure an equal comparison between the various stocks of beets this variety was again planted next day near the center of the field. All other varieties were planted on April 18, in rows east and west 20 inches apart and to an average depth of three-fourths to 1½ inches, with a regulation "shoe" beet drill with the press wheels on, set about three-fourths of an inch apart, so that the soil was not compacted immediately over the seed, but only on each side of the row. There was planted just two drill widths of each variety or stock, making eight rows of each.
>
> Seed was planted at the rate of about 22 pounds per acre, the idea being to plant enough seed to make a full stand of beets. The beet seed evidently appreciated the good treatment it had received; for on the ninth day a few were coming up, and on April 28 the young plants were just breaking the crust. On May 2 every row was showing distinctly, with no difference whatever in the rate of germination between the various stocks of seed.
>
> Thinning the beets to a distance of 8 inches apart was begun on May 29, and finished on June 6. At the beginning the young plants had an average of 7 leaves to a plant and at the last the beets had increased to an average of 12 leaves to the plant.
>
> When the beets had attained some size cultivation was begun, the first being given on May 22 with the beet cultivator. As a rule after the beets had become of some size a furrow was made between the rows at the time of cultivating to allow of irrigation later. The water used in irrigating was not measured, but from previous experience a close estimate is believed to be about 0.3 or 0.4 foot over the ground at each period. The water was allowed to flow between the rows until the soil was thoroughly saturated.
>
> The following notes were made in the field at the time:
>
> *May 22.*—Cultivated with beet cultivator for the first time.
>
> *May 24.*—The young plants are growing very rapidly, but some roots show an attack of a fungous disease like "damping off." The roots of some of the young plants are dead and shriveled and others partly decayed. It is believed this disease may be due to the fungous disease "rhizotonia," which pervades our soils in this locality.
>
> *June 10.*—Cultivated and furrowed for irrigation.
>
> *June 12.*—Irrigating the beets; the water ran in the furrows an average of twelve hours each; some furrows had water as long as forty-eight hours. The beets are doing well and look exceedingly thrifty. A fine stand, none missing. An inspector from a sugar factory said it was the best stand on a small area in the neighborhood.

June 18.—Cultivated.

June 19.—Weeds hoed out and ground cleaned by hand.

June 27.—The beets are exceedingly thrifty, almost covering the ground; hardly a weed in evidence.

July 24.—Cultivated and furrowed for irrigation at the same time. The soil is very moist from recent rains.

July 26, 27, 28.—Irrigated thoroughly with small head of water. Water is very scarce just now. This is the last irrigation the beets can receive.

July 31.—Weeded by hand.

On September 12 an early frost damaged the leaves to such an extent that the selection of beets for mother beets, from the character of the leaves, was suspended for a time. The leaves began to grow again in a few days, and in a couple of weeks an entire new crop of leaves was out, when the selection was continued. The recovery of growth was helped greatly by the downpour of rain on September 20–21, amounting to nearly 6 inches. It was feared at first that this heavy rain would retard the ripening of the beets or lessen the sugar content considerably. That this did not take place to any great extent is shown by the analysis of samples of these beets taken from one of the plats during the harvesting season and analyzed at the Bureau of Chemistry. The results obtained on all the samples sent to Washington for examination are shown in the following table:

Agricultural and analytical data on beets grown at Fort Collins, Colo.

Date received.	Average weight after topping.	Estimated yield per acre.	Sugar in juice.	Sugar in beet.	Purity.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 24	18.2	20.2	14.0	12.9	80.5
October 7	18.3	26.0	13.1	12.1	78.3
October 9	19.8	....	11.4	10.5	74.0
October 21	22.4	....	10.6	9.8	66.6
October 28	16.5	20.2	14.4	13.2	81.4
November 1	21.4	....	15.5	14.3	83.8
November 11	21.4	31.7	15.1	13.9	80.4
November 14	19.5	24.3	16.0	14.7	87.0
November 22	15.2	28.1	15.6	14.4	84.3
November 29	19.5	28.5	14.9	13.7	78.0
December 6	20.9	26.7	14.8	13.6	79.1
December 13	23.3	20.7	14.1	13.0	79.7
Average	19.7	24.0	14.1	13.0	79.4

The general yield of sugar beets in northern Colorado this season was not as satisfactory as it might have been, the average yield being nearly 9 tons per acre. A close study and measurement of this field showed that the stand of beets could have been at least 10 per cent better, there being one beet out of every ten missing, or an open space in which a beet should have been growing. This was general over the field and not confined to any one variety.

The meteorological conditions under which these beets were grown are given in detail below, Cheyenne, Wyo., 40 miles northeast of Fort Collins, being the nearest station at which the sunshine data were available. In general the weather was too dry for beets. At the beginning of the ripening season, however, the event most worthy of note was the heavy September rain and the general wet weather prevailing for some weeks thereafter.

Meteorological data for Fort Collins, Colo.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	°F.	Inches.	Hours.	Hours.			
May	56.1	2.13	....	....	....	....	....
June	63.6	2.43	....	....	....	....	....
July	66.0	1.31	....	....	....	....	....
Average and total	61.9	5.87	....	....	....	....	....
August	68.7	.67	....	....	....	....	....
September	56.8	7.12	....	....	....	....	....
October	48.8	1.15	....	....	....	15	8
Average and total	58.1	8.94	....	....	....	49	17
General average and sum total	60.0	14.81	....	....	....	94	32

Meteorological data for Cheyenne, Wyo.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	°F.	Inches.	Hours.	Hours.			
May	52.8	2.51	245.9	449.1	55.0	7	12
June	61.0	1.55	266.3	451.9	59.0	7	12
July	63.8	1.49	295.2	458.6	64.0	10	7
Average and total	59.2	5.55	....	....	59.3	24	31
August	67.0	0.53	261.0	427.4	61.0	9	7
September	54.0	3.52	273.9	374.0	73.0	14	6
October	47.8	0.52	218.2	343.9	63.0	11	10
Average and total	56.3	4.57	....	....	65.7	34	23
General average and sum total	57.8	10.12	....	....	62.5	58	54

In the data from Colorado we have a remarkable contribution to the study of the effect of environment upon composition. The total rainfall for the six growing months was 14.81 inches, only 2 or 3 inches below the average precipitation for the nonirrigated stations. The distribution, however, was somewhat unfavorable, as nearly one-half the total quantity fell during the month of September. During the dry months the deficiency in rainfall was corrected by irrigation. The temperatures were low compared with those of the other irrigated areas, the average for the six months being only 60°. There was a large percentage of clear days, and it is evident that the beets received an abundance of direct sunshine.

The yield per acre was very heavy, being nearly five times that obtained at the Pomona, Cal., station, and two and a half times that of the nonirrigated station at Chino. The percentage of sugar in the beet is reasonably satisfactory, but the purity is somewhat too low. It is stated in the report of Mr. Danielson that in richness the beets in northern Colorado were not up to the standard of previous years. With the large yields obtained, however, the yield of sugar must be regarded as highly satisfactory.

THE UTAH STATION.

The following cultural and analytical data have been reported by Director Widtsoe of the Utah station:

The beet plat was one-twentieth of an acre in area. Barnyard manure was applied at the rate of 20 tons per acre during the winter of 1901–02. The plat was plowed 10 inches deep and subsoiled 8 inches deeper on May 12, 1902. It was harrowed immediately afterwards and seeded on May 14, at the rate of 15 pounds of seed per acre, the rows being 20 inches apart. The beets were thinned and cultivated on June 14, and were irrigated on June 11 and on August 14, being cultivated two days after each irrigation. On August 2 the crop was reported to be looking well, with a medium stand, and tops 14 inches high. The irrigation water was not accurately measured, but was not far from 7 inches in depth over the whole plat at each irrigation.

Agricultural and analytical data determined at Logan, Utah.

Date of harvest.	Average weight of beets.	Yield per acre.[a]	Sugar in juice.	Sugar in beet.[b]	Purity coefficient.
1902.	*Ounces.*	*Tons.*	*Per cent.*	*Per cent.*	
September 25	11.48	13.8	15.01	13.8	80.26
September 30	12.08	12.4	13.50	12.4	76.70
October 6	9.87	11.6	14.04	12.9	81.15
October 13	13.20	10.9	14.82	13.6	86.66
October 28	18.59	23.5	15.40	14.2	77.38
Average	13.0	14.4	14.6	13.4	80.43

[a] Approximate estimate made at Washington, D. C.

[b] Calculated at Washington.

Only the temperature and precipitation records were available for Logan, and in connection with these a full set of meteorological data for Salt Lake City, about 66 miles south of the experiment station, is given.

Meteorological data for Logan, Utah.

Month.	Mean temperature.	Precipitation.
1902.	° *F.*	*Inches.*
May	55.5	2.19
June	65.4	.74
July	68.4	.52
Average and total	59.8	3.45
August	70.5	.27
September	60.4	Trace.
October	41.4	.51
Average and total	60.8	.78
General average and sum total	60.3	4.23

Meteorological data for Salt Lake City, Utah.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
1902.	° *F.*	*Inches.*	*Hours.*	*Hours.*			
May	59.2	0.33	308.0	449.1	69	15	6
June	69.4	.37	367.1	451.9	81	19	2
July	72.5	.56	372.4	458.6	81	22	4
Average and total	67.0	1.26	....	....	77	56	12
August	74.6	.15	330.4	427.4	77	19	3
September	64.1	.05	302.4	374.0	81	20	5
October	55.2	.52	280.6	343.9	82	21	5
Average and total	64.6	.72	....	....	80	60	13
General average and sum total	65.8	1.98	....	....	78.5	116	25

The results of the collaborative experiments at Utah show the production of a crop of about average tonnage, or at least what the average tonnage should be, a fair content of sugar, and a purity which reaches the standard for successful and profitable sugar manufacture.

The average temperature of the six growing months was 60.3°. August was the warmest month, its average temperature being 70.5°, while October was quite cool, having an average temperature of 41.4°. The precipitation was deficient except for the month of May, and the total amount of rainfall was only 4.23 inches for the six months.

GENERAL REMARKS.

The data from the irrigated sections are interesting in many respects as compared with those from the stations where irrigation is not practiced. In the first place it is evident that the extremes of heat which are experienced in the nonirrigated areas are avoided at the irrigated stations where the temperature is more equable, less prone to reach extreme maxima, and thus should be favorable, according to the results of our investigation, to securing a high content of sugar. And the figures show that the average percentage of sugar in the beets from the irrigated areas is 2.4 per cent higher than that of the other stations.

There was a marked deficiency in the rainfall at the California stations, while in Colorado the total amount of rain would have been sufficient to produce a fair crop had it been properly distributed throughout the growing season instead of one-half of it falling in a single month, namely, September, when it would probably be detrimental to the maturing of the beet.

By reason of the high altitudes there is danger of unseasonable lowering of the temperature, as was shown especially in Colorado, where severe frosts sufficient to check growth occurred in September. It is evident that latitude abstractly has little to do with the climatic conditions which prevail in most of the arid areas because by reason of the influence of the sea on the western coast and the high altitudes of the plateaus, which form the greatest part of the surface of the arid States, the isothermal lines are greatly disturbed in their relation to the parallels of latitude. We have thus introduced into the study of these stations a seriously disturbing factor, the influence of which must be determined by further investigation. The more extensive data which we hope to receive during the next two years from these irrigated areas will form a basis for the solution of this important question.

As only three of the irrigation stations took part in the experiment and the work has only just been systematically inaugurated no attempt is made to represent the data collected in graphic form.

THE SOILS.

The following data, reported by the cooperating stations in regard to the soils on which the beets were grown, are submitted, although they are of rather a fragmentary character. Nevertheless, they are of interest in connection with the chemical and mechanical analyses of such samples as were received for 1902 and the data obtained in 1901.[a]

DESCRIPTIVE NOTES ON SOILS.

UNIRRIGATED SOILS.

AGRICULTURAL COLLEGE, MICH.

[Nos. 25106 and 25107.]

Director Smith sent the following notes on the sampling of soils at the Michigan station:

> The experimental plat is from the center of a larger plat which has borne no crop at all since 1900. We made an attempt to get the soil sample from another part of the plat, but found the conditions so irregular that we had to go to another part of the area. Here we found the soil proper to be but 6 inches deep, the depth of the plowing for the years the plat has lain fallow. The subsoil was rather irregular, as it is bound to be here in the glacial region. A few years ago I made a topographical survey of certain plats on this area on which the varying yields of contiguous plats had indicated some peculiar conditions, and found that the subsoil rose and fell in waves, the crests of which were approximately a rod apart with a height of 6 feet.
>
> The experiments were made on Nos. 33 and 35 of the station plats. These plats are 8 rods long and 2 rods wide. A subplat 60 feet on each side was laid off on the southern part of the plat, the south line being approximately the south line of the plats and the east line of plat No. 33. From the center of this plat a sample of soil was taken for analysis at Washington. From 1891 to 1899, inclusive, oats were grown on these plats with the exception of the years 1893 and 1899, when wheat was planted.

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[a] U. S. Dept. Agr., Bureau of Chemistry Bul. 74, The influence of soil and climate upon the composition of the sugar beet.

ITHACA, N. Y.

[Nos. 24898 and 24899.]

The soil used for this year's experiment is a gravelly loam. It lays at the foot of a knoll and is naturally more fertile than the average soil of the field, but it has had no manure for several years.

GENEVA, N. Y.

[Nos. 1754 and 1755 S. and F].

The soil on which the beets were grown is a clay loam used in a five-year rotation, as follows: 1897, oats; 1898, wheat seeded to clover and timothy; 1899, clover and timothy, first cutting; 1900, clover and timothy, second cutting; 1901, Indian corn or maize.

BLACKSBURG, VA.

[Nos. 25104 and 25105.]

The character of this soil, its fertilization, etc., have been described under the agricultural data given for the Virginia station. The detailed account of the taking of the soil samples as forwarded by Mr. Alwood is as follows:

Opening No. 1.—The top soil, 10 inches deep, grayish, dark, almost slate colored, shows many pebbles and a considerable quantity of humus present. Soil is very mellow down to the subsoil. The line of demarcation between the soil and the subsoil is very distinct. The subsoil at this place is an ochreous yellow, decidedly clayey in texture, and quite moist, firm, but not hard.

Opening No. 2.—The top soil, 12 inches deep, had the same color and appearance as the above; very mellow, humus matter quite abundant, line of demarcation very distinct. Subsoil, gray in color and of very fine texture, was compacted into a tight hardpan, extremely dry and very hard.

Opening No. 3.—The soil was the same as No. 2, 12 inches deep. The subsoil was still harder than No. 2, very dry and with the same general color.

The season was extremely dry and it appears that the ground water failed to rise through the subsoil at the last two openings. At the first opening the ground water was very evident in the subsoil.

MADISON, WIS.

[Nos. 25108 and 25109.]

The soil is a clay loam with a heavy clay subsoil. It has a strong tendency to bake after rains, a thick solid crust being then formed on the surface.

IRRIGATED SOILS.

POMONA, CAL.

At Pomona station the sugar-beet soil is a sandy loam, free from alkali. The chemical character of the soil is the same as that of the Pomona station tract in general, except that it is more diluted from the admixture of a larger proportion of sand, thus diluting the plant food to some extent. An analysis of the soil from the beet plat is as follows:

	Per cent.
Coarse materials > 0.55 mm	33.00
Fine earth	66.00
Chemical analysis of fine earth:	
Insoluble matter	68.40
Soluble silica	10.29
Potash (K_2O)	.89
Soda (Na_2O)	.42
Lime (CaO)	2.53
Magnesia (MgO)	1.84
Oxid of manganese (Mn_3O_4)	.02
Peroxid of iron (Fe_2O_3)	7.13
Alumina (Al_2O_3)	4.41
Phosphoric acid (P_2O_5)	.23
Sulphuric acid (SO_3)	.03
Carbonic acid (CO_2)	.08
Water and organic matter	3.21
Total	99.48
Humus	.58
Humus ash	.40
Humus nitrogen (per cent in humus)	1.16
Soluble potash (citric acid method)	.058
Soluble phosphoric acid (citric acid method)	.05

At Chino station (unirrigated) the soil is somewhat heavier in character than at Pomona, and more retentive of moisture as well as more fertile. The tract has been subdivided and in each subdivision the alkali content has been determined.

FORT COLLINS, COLO.

[Nos. 25060 and 25061.]

The following comments on the soil conditions existing at the Colorado station are taken from the report made by Mr. Danielson:

The ground on which these beets were grown was planted to potatoes the previous season (1901). The potatoes had received no water and only one cultivation. On the west side of the plats the potatoes had been fertilized with bone meal and nitrate of soda in strips running north and south. The beets were planted across these fertilized strips, so as to give all the varieties uniform treatment.

A mass analysis is given of soil from the station farm from a field not far distant from the one on which these beets were grown, and probably very similar. The analysis was made by W. P. Headden.

	Per cent.
Silicic acid	69.356
Sulphuric acid	.041
Carbonic acid	.016
Chlorin	.006
Phosphoric acid	.466
Potash	2.248
Soda	1.215

	Per cent.
Lime	1.645
Magnesia	1.412
Ferric oxid	5.424
Manganic oxid	.160
Moisture at 110° C	2.981
Ignition	4.044
	100.352
Oxygen eq. chlorin	.001
	100.351

Samples of the soil and subsoil taken in the middle of the growing season from the area on which these beets were grown were forwarded to Washington.

ANALYSES OF SOILS.

The chemical and mechanical analyses of the samples of soils and subsoils forwarded to Washington from the various stations were made in the Bureau of Chemistry and in the Bureau of Soils of this Department, respectively, and the results are shown in the following table:

Chemical analyses of sugar-beet soils, 1902.

[Percentages based on water-free soil.]

Serial No.	Locality.	Description.	Insoluble.	Volatile.	Nitrogen (N).	Soluble in 1.115 sp. gr. HCl.					Soluble in N/200 HCl. Parts per million.	
						Potash (K_2O).	Lime (CaO).	Magnesia (MgO).	Fe_2O_3, Al_2O_3, Mn_3O_4.	Phosphoric acid (P_2O_5).	Phosphoric acid (P_2O_5).	Potash (K_2O).
			P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*		
25100	Lexington, Ky	Soil	80.96	8.25	0.24	0.28	0.75	0.40	8.48	0.75	7.0	51.6
25101	do	Subsoil.	85.20	5.58	.14	.08	.59	.35	7.44	.50	......	......
25106	Agricultural College, Mich.	Soil	89.40	4.85	.11	.25	.62	.36	4.50	.09	2.0	37.0
25107	do	Subsoil.	92.79	2.15	.03	.28	.38	.39	3.20	.03	......	......
24898	Ithaca, N.Y. (Cornell).	Soil	87.58	7.30	.17	.21	.35	.45	3.09	.18	10.7	114.0
24899	do	Subsoil.	83.74	4.00	.06	.66	2.00	.84	8.03	.09	......	......
1754 (S. & F.)	Geneva, N. Y.[a]	Soil	81.70	8.00	.17	.56	.56	.82	8.34	.09	1.0	87.0
1755 (S. & F.)	do	Subsoil.	80.35	5.60	.10	.89	.81	1.27	10.91	.09	......	......
25104	Blacksburg, Va	Soil	87.03	4.32	.17	.30	.19	.38	6.67	.14	5.0	249.6
25105	do	Subsoil.	87.30	2.90	.03	.36	.16	.47	8.62	.07	......	......
25108	Madison, Wis	Soil	85.76	5.80	.13	.38	.61	.45	5.52	.16	26.0	48.0
25109	do	Subsoil.	85.30	5.00	.06	.44	.46	.67	8.00	.11	......	......
25125[b]	Washington, D.C.	Soil	83.47	5.35	.18	.39	.47	.51	8.27	.03	......	......
	Irrigated soils.											
25060	Fort Collins, Colo	Soil	73.01	10.20	.18	.73	5.40	1.11	9.01	.18	3.5	144.6
25061	do	Subsoil.	65.39	12.50	.11	.54	10.16	1.27	9.35	.17	......	......
1756 (S. & F.)	Logan, Utah[c]	Soil	78.46	13.50	.10	.73	6.82	4.34	6.27	.22	25.0	394.2

[a] Samples taken after crop was grown.

[b] Analysis made in 1901. Same plat planted in 1902. Soil only analyzed.

[c] No subsoil sent, as plat is very shallow and underlaid with gravel.

Mechanical analyses of sugar-beet soils, 1902.

Serial No.	Locality.	Description.	Organic matter.	Gravel, 2 to 1 mm.	Coarse sand, 1 to 0.5 mm.	Medium sand, 0.5 to 0.25 mm.	Fine sand, 0.25 to 0.1 mm.	Very fine sand, 0.1 to 0.05 mm.	Silt, 0.05 to 0.005 mm.	Clay, 0.005 to 0.0001 mm.
			Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
25100	Lexington, Ky	Soil	3.08	0.06	1.30	1.36	1.04	1.76	75.90	18.58
25101	do	Subsoil	1.43	0.00	.98	.50	.56	1.24	76.72	19.10
25106	Agricultural College, Mich	Soil	2.58	2.30	4.48	9.16	33.96	14.36	27.12	8.62
25107	do	Subsoil	.54	1.14	4.84	9.80	32.80	16.88	21.06	13.48
24898	Ithaca, N.Y. (Cornell).	Soil	3.42	.60	5.80	6.50	27.20	24.84	20.30	14.88
24899	do	Subsoil	1.34	0.00	3.08	3.90	31.06	34.00	17.46	10.26
1754 (S. & F.)	Geneva, N. Y	Soil	2.22	4.88	4.36	3.36	10.26	17.98	29.84	29.30
1755 (S. & F.)	do	Subsoil	.68	3.10	4.40	3.50	9.80	16.80	27.10	35.02
25104	Blacksburg, Va	Soil	1.82	3.56	3.52	2.44	6.58	10.10	58.62	15.18
25105	do	Subsoil	1.23	3.68	3.00	2.36	5.70	8.84	55.64	20.78
25108	Madison, Wis	Soil	3.14	0.00	1.30	2.20	7.40	6.40	65.50	16.74
25109	do	Subsoil	.78	0.00	.80	1.40	5.62	6.00	65.60	20.56
25125[a]	Washington, D.C.	Soil	1.63	.34	1.52	2.94	16.78	24.00	33.02	21.40
	Irrigated soils.									
25060	Fort Collins, Colo	Soil	2.52	.08	3.28	3.14	13.92	24.26	36.12	19.20
25061	do	Subsoil	1.09	.06	2.28	1.98	9.48	22.20	38.86	24.90
1756 (S. & F.)	Logan, Utah	Soil	5.83	1.10	1.60	1.94	22.80	35.16	22.58	14.80

[a] Analysis made in 1901. Same plat planted in 1902. Soil only analyzed.

COMMENT ON ANALYSES.

The data of the soil analyses are calculated to the water-free substances with the exception of water of composition. A general comparison of the soils of the irrigated and nonirrigated stations shows a larger percentage of insoluble matter in the first named than in the latter. On the other hand, the irrigated soils show a larger percentage of volatile matter than the nonirrigated. It is evident that the irrigated soils are more highly basic and the nonirrigated soils more highly acidic in character. The irrigated soils have a much higher average percentage of potash than the nonirrigated, and, with the exception of the soil from Lexington, they have a higher average content of phosphoric acid. The most marked difference, however, in the two sets of soils is in the quantity of lime contained, the irrigated soils showing a very much larger proportion of this important element. This is easily explained when it is remembered that lime compounds, especially the carbonate, are quite soluble in water carrying carbon dioxid, and thus the lime is more thoroughly leached from rain washed, nonirrigated soils. The large quantities of lime and potash present in the irrigated soils can not fail to be of lasting benefit to such a crop as the sugar beet. Potash furnishes a most important part of the food of the beet, while the lime tends to favor the conditions producing maximum nitration of the organic nitrogenous bodies which the soil may contain.

The irrigated soils, when treated with dilute cold hydrochloric acid, show larger quantities of potash than the nonirrigated, with the exception of the soil from Blacksburg, Va. The latter soil, although it does not have a very large percentage of potash, evidently holds it in a state easily assimilated by growing crops.

The nitrogenous content of the soils is quite uniform, but in some of the subsoils, as for instance those from Michigan and Virginia, the content of nitrogen is very low. Phosphoric acid is not very abundant in any of the soils except that from Lexington, Ky., which is well known as a carrier of large quantities of this important plant food.

The mechanical composition of the soil shows some interesting variations in the distribution of the materials of different degrees of fineness. The soil from the Lexington station is found to be three-fourths silt while that from the Ithaca station is only one-fifth silt. The soil from the Geneva station, where the richest beets are produced, is composed of almost one-third silt. In general it may be said that so large a proportion of silt as is found in the Lexington soil would tend to produce a seed bed somewhat too compact for the development of a beet of the best quality. Yet we find in the soil from Madison, Wis., that the percentage of silt is only 10 per cent less than that at Lexington. The yield of beets, however, at Madison and the content of sugar therein were both extremely satisfactory.

The organic matter varied greatly in the different soils, being present in the largest quantity in the sample from Logan, Utah, and the smallest in the soil from Washington, D. C., the latter being an artificial soil made from the materials dredged from the bottom of the Potomac River. Among the subsoils the one having the least organic matter was that from the Michigan station and the highest content was found in the sample from the Kentucky station.

SUMMARY OF DATA.

Agricultural and analytical data, 1902.

Station.	Mean weight of topped beets.	Estimated yield per acre.	Sugar in beet.	Coefficient of purity.
	Ounces.	*Tons.*	*Per cent.*	
Lexington, Ky	8.0	8.9	7.3	70.9
Washington, D. C	22.9	26.1	8.4	72.4
Blacksburg, Va	15.4	[a] 16.7	11.7	74.4
Ithaca, N. Y	17.0	18.0	12.5	81.9
Madison, Wis	24.2	31.8	12.7	82.0
Agricultural College, Mich	10.6	12.5	13.5	86.9
Geneva, N. Y	14.3	16.1	13.9	84.5
Irrigation stations.				
Fort Collins, Colo	19.7	24.0	13.0	79.4
Logan, Utah	13.0	14.4	13.4	80.4
Pomona, Cal		5.0	15.0	86.5

[a] Estimated on 50 feet of row harvested September 1 and 19.

Yield and soil data, 1902.[a]

Station.	Yield per acre.	Chemical analysis.			Mechanical analysis.		
		Potash.	Nitrogen.	Phosphoric acid.	Total sand.	Silt.	Clay.
	Tons.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Lexington, Ky	8.9	0.18	0.19	0.63	4.37	76.31	18.84
Agricultural College, Mich	12.5	.27	.07	.06	63.14	24.09	11.05
Geneva, N. Y	16.1	.73	.14	.09	35.23	28.47	32.06
Blacksburg, Va	16.7	.33	.10	.11	21.27	57.13	17.98
Ithaca, N. Y	18.0	.44	.12	.14	68.19	18.88	12.57
Washington, D. C.[b]	26.1	.39	.18	.03	45.24	33.02	21.40
Madison, Wis	31.8	.41	.10	.14	15.56	65.55	18.65
Irrigated soils.							
Pomona, Cal.[c]	5.0	.89	....	.23	....	....	....
Logan, Utah[d]	13.0	.73	.10	.22	61.50	22.58	14.80
Fort Collins, Colo	24.0	.64	.15	.18	40.27	37.49	22.05

[a] Average of figures for soil and subsoil as platted on charts are given.
[b] Soil only for 1901.
[c] Determined at California station; only humus nitrogen given.
[d] Soil only.

Meteorological data, May to October, 1902.

Station.	Temperature.	Precipitation.	Clear days.	Cloudy days.	Sunshine.
	°*F.*	*Inches.*			*Per cent.*
Lexington, Ky	69.3	16.6	83	22	76.1
Washington, D. C	68.6	23.5	80	25	67
Blacksburg, Va	65.8	15.2	74	46	....
Ithaca, N. Y	60.4	23.3	41	54	....
Madison, Wis	61.8	24.6	38	76	....
Agricultural College, Mich	60.5	27.4	55	67	[a]58
Geneva, N. Y.[b]	63.1	20.2	....	....	....
Irrigation stations.					
Fort Collins, Colo	60.0	14.8	94	32	[c]62.5
Logan, Utah	60.3	4.2	[d]116	[d]25	[d]78.5
Pomona, Cal	70.0	.59	67	6	[e]70

[a] Sunshine data for Detroit, Mich.
[b] Data for May observed at Lyons.
[c] Sunshine data for Cheyenne, Wyo.
[d] Report for Salt Lake City, Utah.
[e] Report for Los Angeles, Cal.

Geodetic data.

Station.	Average length of day.[a]		Latitude.[b]			Altitude.[b]
	h.	*m.*	°	′	″	*Feet.*
Lexington, Ky	14	18	38	02	25	979
Washington, D. C	14	23	38	53	23	37.5
Blacksburg, Va	14	14	37	14	00	2,100
Ithaca, N. Y	14	11	42	27	00	810
Madison, Wis	14	44	43	04	36	955
Agricultural College, Mich	14	42	42	45	00	847
Geneva, N. Y	14	44	42	53	00	458
Irrigation stations.						
Fort Collins, Colo	14	32	40	35	00	4,994
Logan, Utah	14	37	41	44	00	4,506
Pomona, Cal	13	58	34	3	00	861

[a] These figures cover from May to August, inclusive, and are furnished by the U. S. Naval Observatory.
[b] Data furnished by the U. S. Coast and Geodetic Survey.

CONCLUSIONS.

The results of the investigations of 1902 confirm in a general way those of the two previous years. As is to be expected, however, the great seasonal changes which take place from year to year at the different stations introduce important variations in the chemical composition of the crop. For the third time in succession, however, the station at Geneva holds first place in respect of the content of sugar.

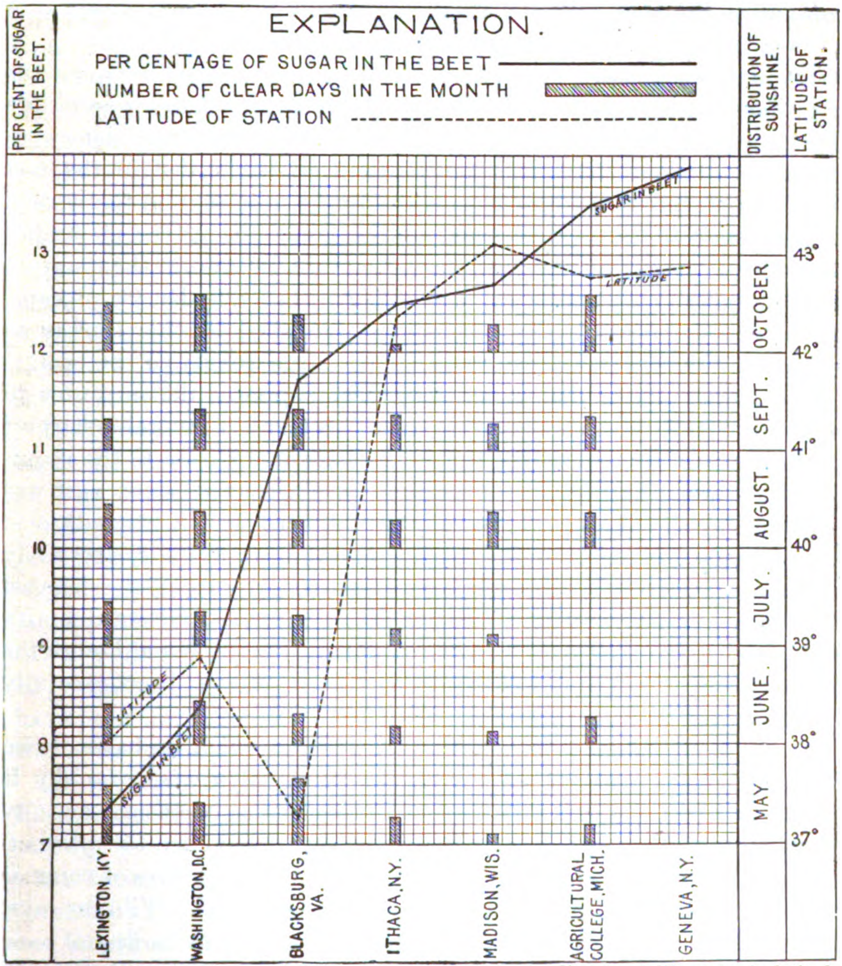

FIG. 1.—Sugar content of beets as influenced by variations in cloudiness and differences in latitude of experiment stations.

Michigan has advanced to second place, after having been tied with the stations of Ithaca and Lafayette in 1901, for that position.

The chart (fig. 1) shows the percentage of sugar in the beet, the number of clear days in the month during the growing season, and the latitude of the station. By reason of the fragmentary nature of the data for the percentage of sunshine the curve representing this element of the environment has been omitted. As in the two previous years, there is a general agreement in the curves showing the percentage of sugar and the latitude. The one important exception is found in the Blacksburg station, and this is fully explained by reason of the high altitude of this locality, which acts in the same manner as a high latitude. Were it not for the varying length of the day a more definite relation could be established by comparing the sugar content of the beet with the mean isothermal lines for the period of growth, instead of with the latitude.

The number of clear days was very evenly distributed between the growing months at Lexington, Washington, and Blacksburg and quite unevenly distributed at the other stations for which these data were reported. In our previous reports attention was called to the fact that unobstructed sunshine is not absolutely necessary to the normal development of the sugar content of the beet.[a] Some recent studies made in Germany on the influence of certain factors of the environment upon the composition and yield of sugar beets, by Dr. O. Vibrans, of Helmstedt,[b] confirm the conclusions published by this Bureau in regard to the effect of cloudy weather upon the quality of the beet.

The author states that it has long been supposed that in order to secure a normal evolution of the sugar beet and a favorable harvest continued warmth and sunshine are necessary in addition to the requisite moisture. The season of 1902, however, characterized as it was by excessive cloudiness and precipitation, showed that these conditions, especially as bearing upon the quality of the beet, are not necessarily detrimental. It is stated, however, that the result of the harvest showed a diminished yield in spite of the fact that a sufficient quantity of moisture was received during the growing season and although the fields were well cultivated, normally fertilized, and the beets carefully watched.

These observations bear out the conclusions which we have drawn from our studies to the effect that the sun's rays when the sky is covered with clouds are still able to influence the functional activity of the chlorophyll cells in such a way as not to diminish the percentage of sugar in the beet. In fact during the very hot days of summer such a screening of the sunlight may prove beneficial. Vibrans says:

> The weakening of the total intensity of the sun's action by the particles of water and dust which may be in the air is greatly increased in the case of clouds. If a cloudy condition continues for some time the sun's rays nevertheless, as has been said, continue to exert their influence on the plant. This has been shown by the peculiar phenomena which were manifested during the past year (1902), when it was

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[a] U. S. Dept. Agr., Bureau of Chemistry Bul. 64, p. 30, and Bul. 74, p. 37.

[b] Centralblatt für die Zucker-Industrie, No. 33, May 16, 1903, p. 809, and No. 34, May 23, p. 829.

noticed that those sugar beets which were particularly rich in sugar had developed an excessive leaf growth. From this the conclusion can be drawn that the leaves which were at first developed by the beet were not sufficient to produce a normal development with the quantity of light at their disposal. The dormant buds, therefore, woke into activity and produced the additional leaves required. Thus a more extended leaf surface was created and the light could thus engender the necessary activity to develop new vital processes.

It is further shown that this increase of leaf surface which permits the assimilation process to go on unhindered in cloudy weather is not noticed in conditions of artificial shade which tend to produce etiolation—as, for instance, on the edges of forests. In such cases the leaf stems are unduly lengthened, thin, and weak, and the leaves do not reach normal size because those processes in the leaf cells which promote the formation of leaf surface do not develop normally. The result of this is that the processes of assimilation and condensation attending the activity of the chlorophyll cells in the formation of starch and sugar are not complete. Greater comparative quantities of nonsugars are formed and the value as well as the quantity of the beets produced is greatly diminished. The size of the beet leaf in relation to the stem is dependent on the quantity and intensity of the light, and the more refractive elements of the sun's rays appear to be most active in producing such growth.

In the case cited by Vibrans as to the effect of artificial shading of beets grown along a road bordered by thick, heavy, chestnut trees, he states that the beets not only had a lower sugar content, but also yielded a decreased tonnage. It appears, however, that Vibrans has probably fallen into some error in ascribing this diminution of both sugar content and tonnage solely to the shade of the trees. It is well known that trees draw upon the adjacent soil for nourishment and to this extent rob any cultivated plant that may be in the vicinity. A large number of the poorer quality of beets in that experiment may have been due, therefore, to the absorbing activity of the rootlets of the trees extending great distances under the growing crop.

On the chart (fig. 2, p. 44) are shown the percentage of sugar in the the beet, coefficient of purity of the juice, the temperature and the average length of day. Here again is seen the X-like figure—somewhat fantastic in shape this year, but still marked in character—formed by the lines representing the content of sugar, purity of juice, and length of day, with the line representing the temperature. The variations in the temperature curve are found chiefly at Ithaca and Geneva. At Geneva, especially during the present year, the temperature of the growing season was high, while at Ithaca it was the lowest of any recorded at the cooperating stations. The highest temperature observed was at Lexington, Ky. The data represented on Chart No. 2 are in general wholly confirmatory of those obtained in the previous years of the investigation.

On the chart (fig. 3, p. 45) are shown the percentage of sugar in the beet, the total rainfall and its distribution by months throughout the growing season, together with the altitude of the station. The most characteristic feature of this chart is the monumental curve showing

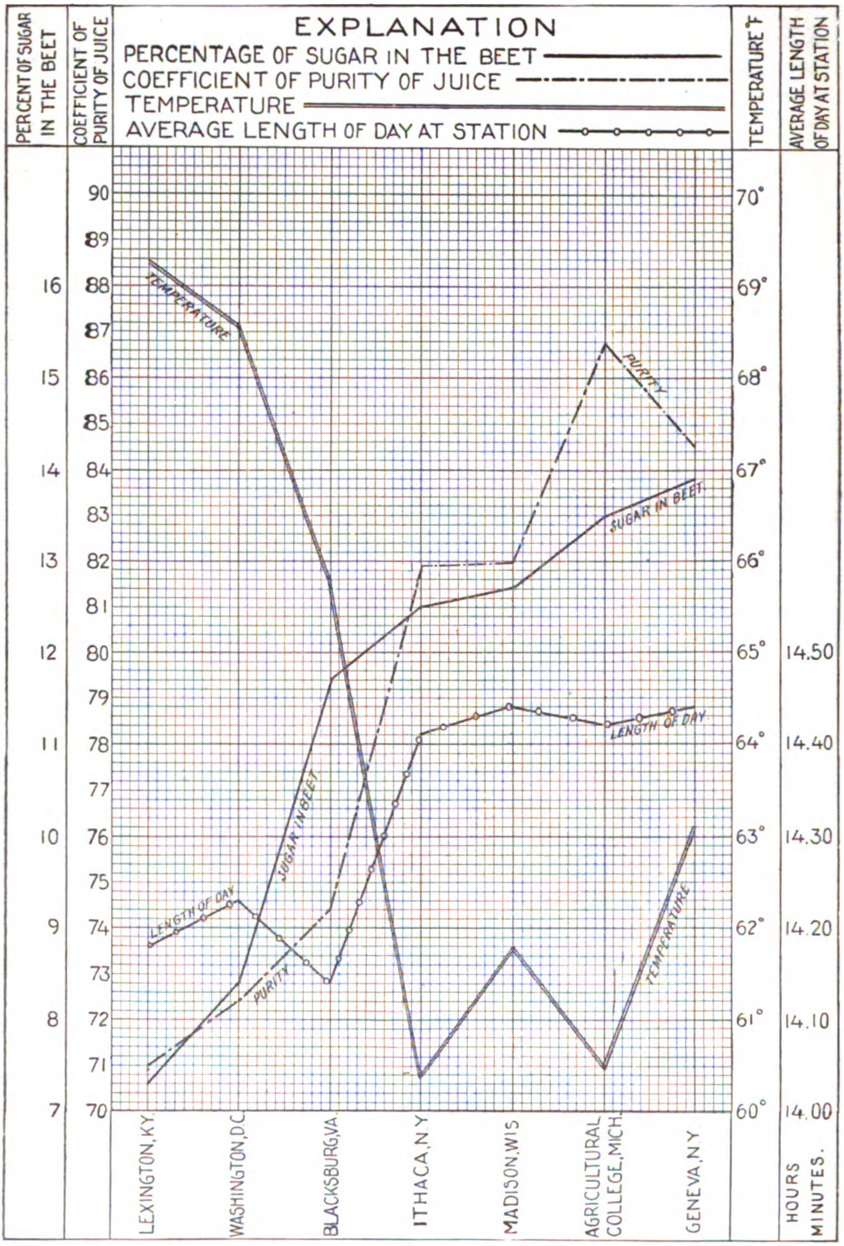

FIG. 2.—Sugar content of beets and purity of juice as influenced by temperature and light.

the altitude of the Blacksburg station. It is evident that altitude can only be considered in connection with the content of sugar in the beet

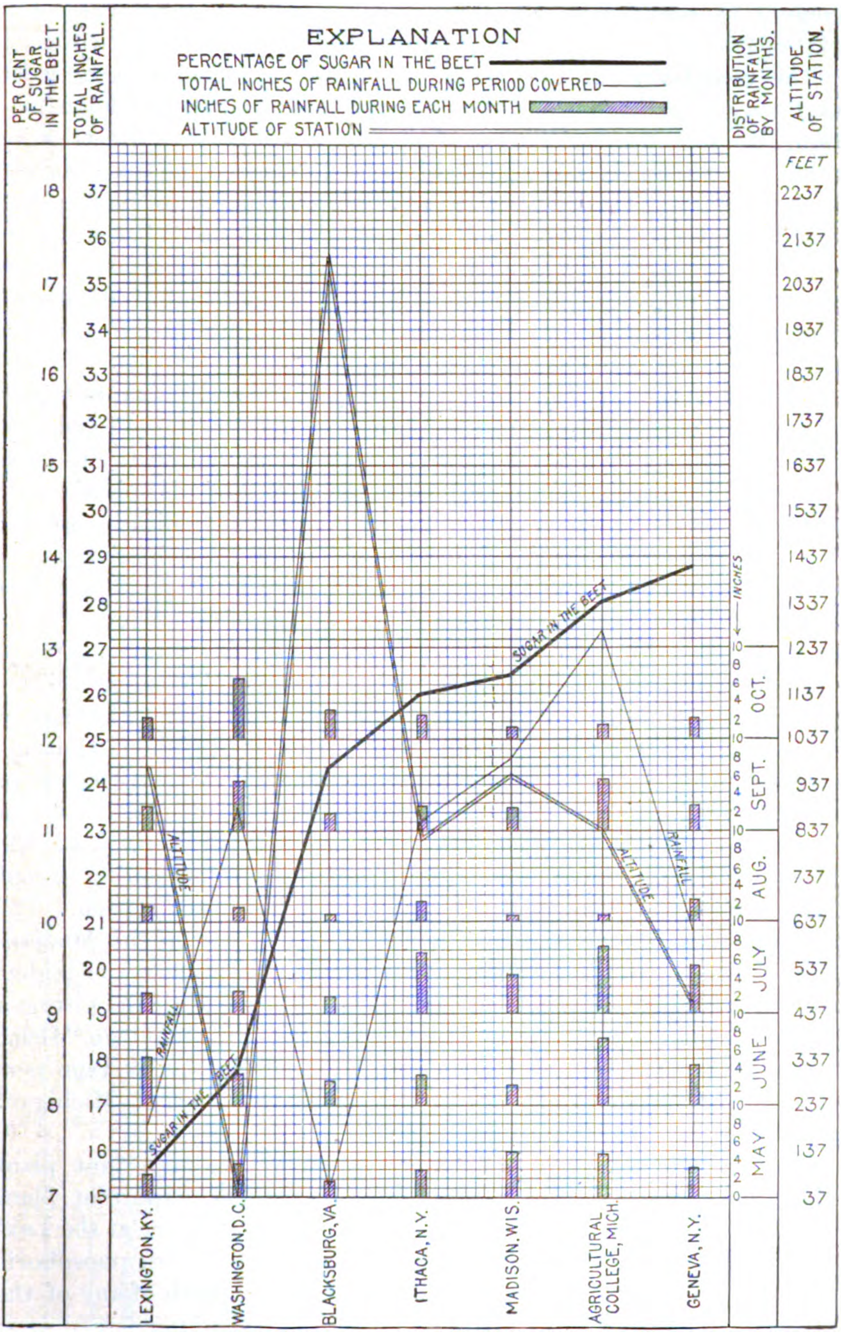

Fig. 8.—Sugar content of beets as influenced by rainfall and altitude of station.

when referred to some fundamental basis of comparison. there is a very marked tendency, however, for the high altitude to counteract the influence of the low latitude. This is shown vividly by the chart in the case of Blacksburg, where by reason of the high altitude the content of sugar was raised far above what would be reasonably expected at sea level or on an extensive plateau at the same latitude.

The distribution of the rainfall is somewhat irregular, being most uniform at Lexington and Geneva, and least so at the Michigan station. In fact, the distribution of the rainfall at Geneva is extremely even, and while it is only reasonably so at Lexington the total amount is satisfactory. This chart gives additional testimony to the fact that under proper conditions of culture, and where the seed bed has been carefully prepared, not only the composition of the beet but also the yield per acre can be considered as largely independent of the distribution of the rainfall, provided there is sufficient moisture to meet the ordinary demands of the growing crop.

The chart (fig. 4, p. 47) shows the yield per acre and the average percentages of potash, nitrogen, and phosphoric acid in the soil and subsoil, the two figures having been combined, as the beets are deep feeders. The determination of potash, nitrogen, and phosphoric acid, according to the methods used, gives only a general indication of soil fertility, though the total absence of any one of these ingredients of plant food would make the production of a crop impossible. The presence, however, of even a small proportion of these foods, reckoned in per cent, indicates a sufficient quantity for many successive crops, if rendered available. The weight of the soil is so great compared with the weight of the crop that is produced upon it that a very few hundredths of a per cent of any one ingredient of plant food means an abundance of this ingredient for the production of the normal crop, provided it is in a form available for plant growth. At the station which had the largest yield per acre, namely, Madison, Wis., the phosphoric acid content was higher than at any other station except Lexington and Ithaca, being the same as at the latter station. The quantity soluble in dilute acid, however, was larger in the Madison than in the Lexington sample. The amount of potash was higher than at any other station except Ithaca and Geneva, while the nitrogen was lower than at any other station except Agricultural College, Mich., being the same as that at Blacksburg, Va. The lowest average percentage of nitrogen at any one of the stations was 0.07 at Michigan, and the highest was 0.19 at Lexington.

The presence of large quantities of two of the important plant foods does not indicate a large crop if a third important plant food is very deficient. This is shown in a marked degree at the Lexington station, which has the highest percentage of both phosphoric acid and nitrogen and the lowest percentage of potash of any of the stations, accompanied by the lowest yield. The yield per acre at Lexington could doubtless be greatly increased by the judicious applica

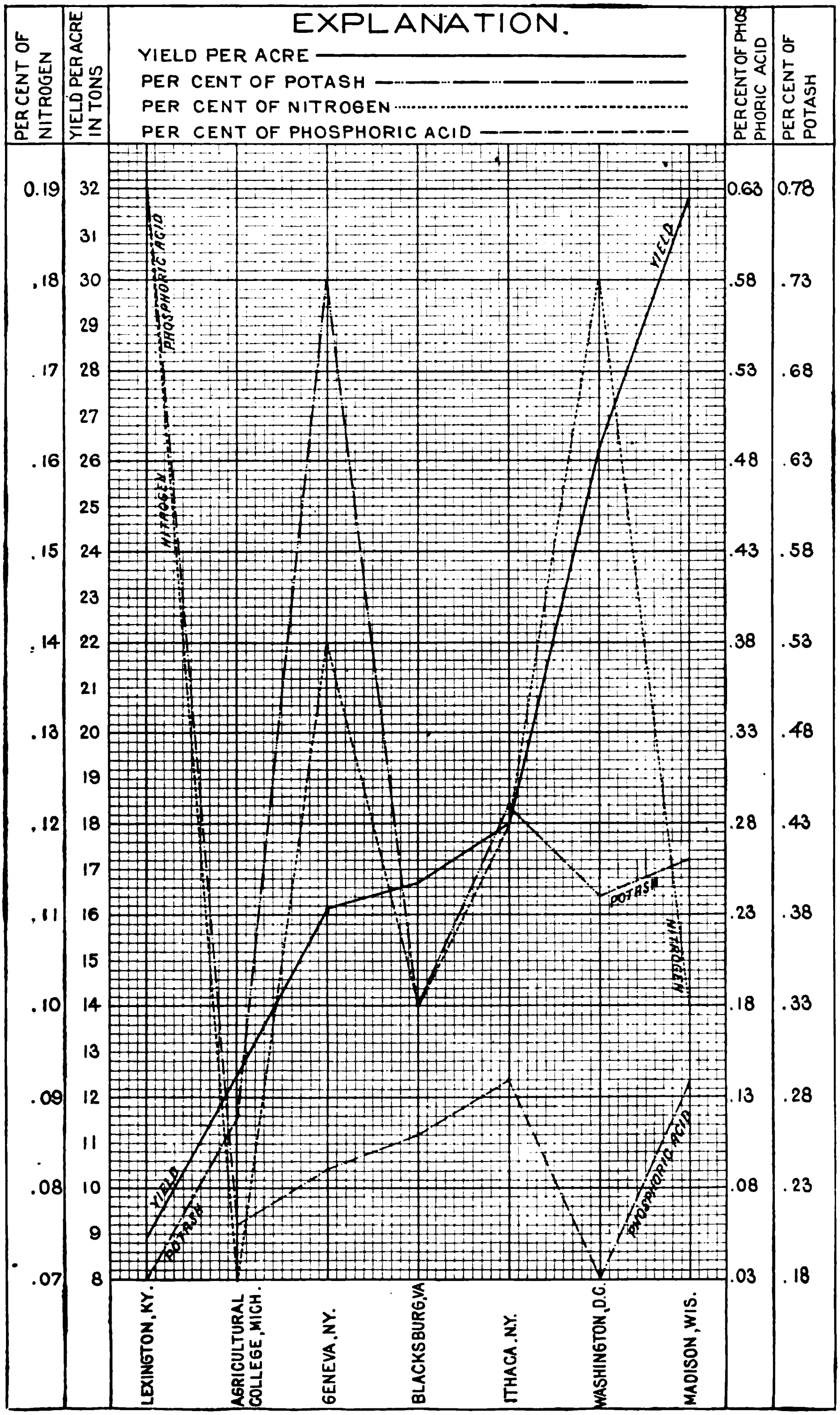

Fig. 4.—Yield of beets per acre as influenced by variations in the nutritive elements of the soil.

tion of potash. Nitrogen must be considered as of a more transitory nature than either potash or phosphoric acid. It exists most abundantly in the soils in the form of organic nitrogen which, though not available for plant food in that form, becomes so under the influence of nitrifying ferments. We have, however, a fine illustration of the influence of the deficiency of two elements of plant food at the Michigan station, where both phosphoric acid and nitrogen were very low, and the yield per acre was next to the lowest given. An apparent contradiction of this is found in the case of Washington, where, although the phosphoric acid was low, the yield was over 26 tons per acre. Both the potash and nitrogen, however, were abundant at the Washington station, and the phosphoric acid, having been derived from the bed of the river, though not large in quantity, was in a form to be readily assimilated. It is evident, however, that the data correlating the quantity of available plant food in a soil and the magnitude of the crop produced have their full value only when the meteorological conditions and all other elements of the environment are the same. This is convincingly illustrated by the results of the pot experiments conducted by this Bureau during the past decade which are now being prepared for publication. The complete failure of a crop on a soil well supplied with the necessary plant foods is described in the report from the Indiana station this year.

The chart (fig. 5, p. 49) shows the yield per acre and the average percentages of total sand, clay, and silt in the soil and subsoil. This chart, in so far as we know, is the first attempt to graphically illustrate the relation of the mechanical composition of the soil to the crop. The problem is so new and the data so fragmentary that we can not claim that much progress has been made in the attempt to elucidate any of the undeveloped principles, if there be such principles, which correlate the mechanical composition of the soil and the yield of the crop. It is evident that the mere mechanical state is not complete evidence of the availability of the plant food, but it is an indication as to whether or not the rootlets of the plant can have access to the nourishment which the soil contains. In general, it is true that the mechanical condition of a soil is highly important in determining the character of the crop which can be grown thereon. For instance, wheat is not the ideal crop for very sandy soils, nor melons and beets for those composed of stiff clay. We have, however, in the soils on which the beets of the present investigation were grown types of almost all kinds of soils, as far as mechanical structure is concerned, and the data obtained illustrate again, as has been developed in the former investigations, that the soil is the least important factor in the environment of the sugar beet in respect of sugar content.

A fundamental condition for the growth of beets is the preparation of the seed bed to a depth of at least 16 inches. Thus, even stiff clay

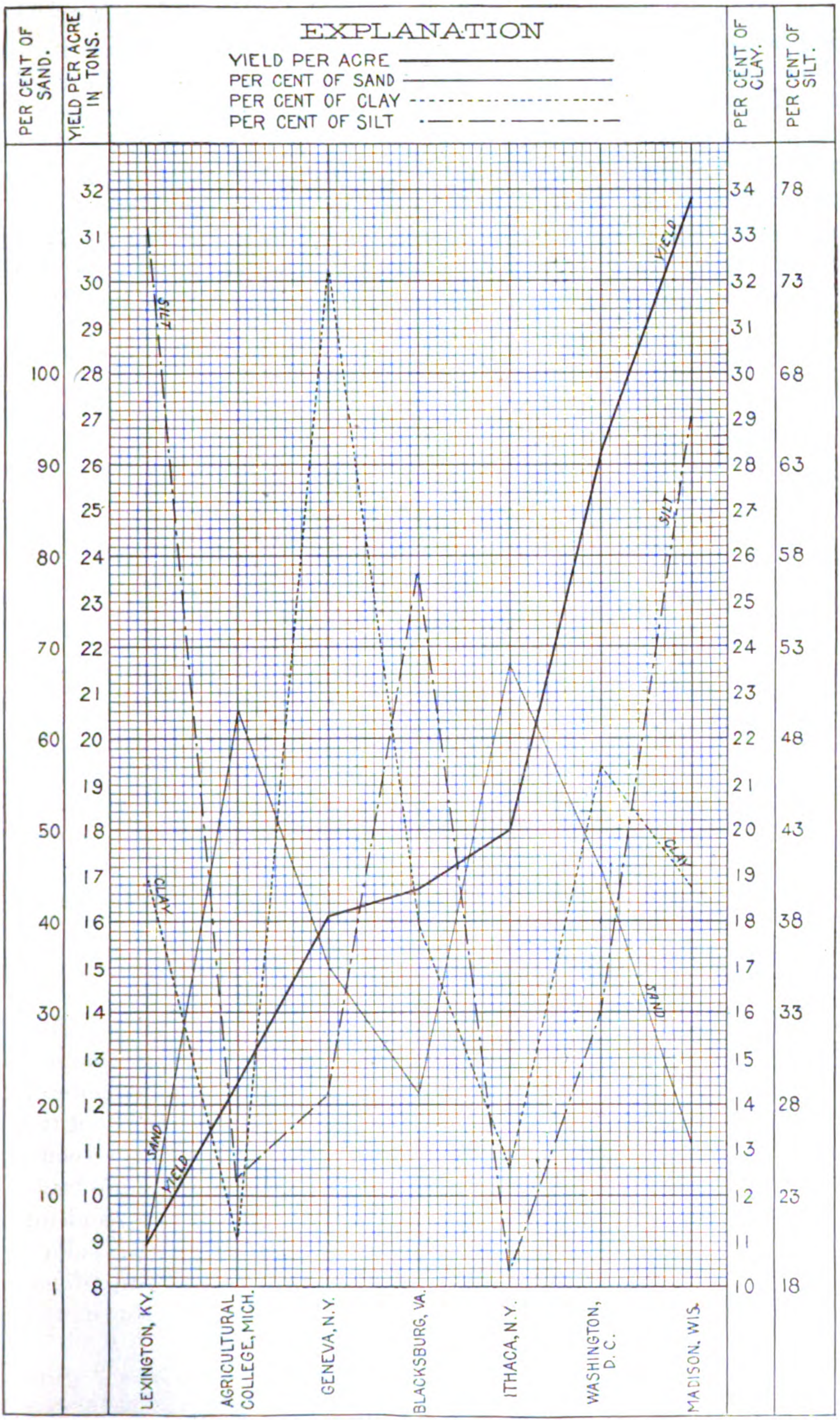

FIG. 5.—Yield of beets per acre as influenced by variations in physical composition of the soil.

when properly prepared is easily permeated by the roots of the growing beets. If a stiff clay soil be not properly prepared, then it is evident that beets could not do nearly so well in it as in a sandy soil; but when both kinds of soil are well prepared the beet grows well in each. It is evident, therefore, that the beet is not well suited to determine the influence of mechanical composition of the soil on the quantity of the crop. While a problem of this kind is very difficult, it is not beyond the possibility of solution, and it is believed that continued collection of data will throw light upon points which, so far as the beet is concerned, must now be considered as very dimly illuminated.

O

U. S. DEPARTMENT OF AGRICULTURE,

BUREAU OF CHEMISTRY—BULLETIN NO. 79.

H. W. WILEY, Chief of Bureau.

THE TESTING OF ROAD MATERIALS,

INCLUDING THE METHODS USED AND THE RESULTS OBTAINED IN THE ROAD MATERIAL LABORATORY

IN COLLABORATION WITH THE OFFICE OF PUBLIC-ROAD INQUIRIES.

BY

LOGAN WALLER PAGE,
Chief, Road Material Laboratory,

WITH THE COOPERATION OF

ALLERTON S. CUSHMAN,
Chemist, Road Material Laboratory.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1903.

LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., July 20, 1903.

Sir: I have the honor to transmit herewith for your approval a manuscript embodying the results of the tests conducted in the Road Material Laboratory of this Bureau since its establishment by you in October, 1900, together with an exposition of the methods used in obtaining these results, and to recommend that this report be published as Bulletin 79 of the Bureau of Chemistry. I beg to make acknowledgment of the cordial cooperation of the Office of Public-Road Inquiries in these investigations, without which this work could not have been done.

The illustrations, which comprise five half-tone photos and ten text figures, are regarded as necessary to a clear understanding of the text.

Respectfully,

H. W. Wiley,
Chief.

Hon. James Wilson,
Secretary of Agriculture.

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THE TESTING OF ROAD MATERIALS.

INTRODUCTION.

The Road Material Laboratory was established by the Secretary of Agriculture in December, 1900. Work was not begun, however, until several months later on account of the time necessary to effect its equipment. The fiscal year ended June 30, 1902, was the first year of its operation. The laboratory at present contains all the necessary appliances for making tests on road materials and for investigating some of the more important problems connected with the work.

The main object of this bulletin is to describe in some detail the methods and work of the laboratory and to give the results obtained up to the present time. Incidentally, the physical, mechanical, and chemical agencies which act upon road materials are discussed and a brief history of the testing of such materials is given.

The Road Material Laboratory tests road materials, without charge, for citizens of the United States and reports to them the information obtained. In addition to routine tests on samples submitted, investigations are conducted for the improvement of road materials. The laboratory is prepared to test rocks, gravels, and clays for road construction, tiling, cement and concrete, and paving blocks of stone, brick, wood, asphalt, and bituminous matter. The principal activities may be classified under the following heads:

(1) Tests for determining the quality of materials to aid road builders in selecting those most suitable for their work.

(2) The investigation of various processes to develop simple, appropriate, and reliable tests.

(3) The collection of data for use in drawing up specifications for standards of quality.

(4) Scientific research to develop new materials or mixtures, and the study of problems which may arise in road building.

The most important work comes under the first head, for the main object of the laboratory is to obtain results, by means of appropriate tests on small samples, which will agree sufficiently well with the results of practice to aid the road builder in selecting the most suitable materials from those available for his work. That the laboratory may serve its purpose, the tests adopted must be of such a nature that they can be executed with dispatch, and the results should be clear and comprehensive. While it is necessary that the results should indicate the wearing qualities of the materials in service, it is unnecessary, as well as impracticable, that an attempt should be made to imitate the exact conditions of service. Laboratory experiments have been designed in various ways to reproduce the conditions that obtain in practice, but they have failed for the obvious reason that the principal element in such conditions is lapse of time, which renders quick results impossible.

It is apparent that no single material or class of materials will suit all conditions of climate and traffic. To get the best results it is necessary that the materials selected, as well as the methods of construction, should be adapted to the special conditions of the road that is to be built or maintained. This is not always practicable and is often impossible. Much can be done, however, by means of laboratory tests to determine which of the available materials is best suited for use, and this work constitutes the chief usefulness of the Road Material Laboratory. A macadam road, when properly constructed, costs from $4,000 to $10,000 a mile, and the cost of many other types of road are greatly in excess of this. An error in the selection of a material means an inferior road and occasionally complete failure. Such an error, therefore, may cause a great loss of money to the community in which it occurs.

No explanation is necessary of the second and third subdivisions of the work, i. e., the development of new tests and the collection of correlated data, which is similar in most respects to that of all testing laboratories.

The fourth subdivision or research work, including the development of new materials, is of a most important character. New road materials are occasionally developed, and the methods for using old ones are constantly improving. It is most important to test and investigate such materials and methods if the best results are to be derived from them. Practically none of our rural towns or counties has the facilities or means to carry on such investigations, and it is in these districts that better roads are most needed. Even when road building is attempted materials and methods are frequently used which are unsuited to the conditions of climate and traffic and the result is failure.

Probably the most difficult problem to solve for the betterment of rural highways, and one on which the Road Material Laboratory has been at work for some time, exists in those sections of our country where no hard materials are to be found from which roads can be constructed. Many of these districts are of very great extent, and have only a clay soil on which and from which to build roads. The result is that during a considerable portion of the year the roads are almost impassable and are rarely good at any time. The Road Material

Laboratory has carefully studied the methods of burning clay for ballast in use by a number of western railroads. By somewhat modifying these methods very satisfactory roads can undoubtedly be made at a moderate cost in districts where the clay is of a suitable nature. Experiments are also being made with mixtures of crude petroleum and asphaltum which give promise of good results.

Before taking up any of the above subjects in detail it will be well to consider first some of the important physical and mechanical properties of road materials, and also some of the principal agents in the destruction of roads.

IMPORTANT PHYSICAL AND MECHANICAL PROPERTIES OF ROAD MATERIALS.

There are three chief properties essential to good road materials, and only these will be considered here. They are hardness, toughness, and cementing or binding power. Although these properties, at least hardness and toughness, have long been recognized by those familiar with the subject, yet they have never been properly defined and the terms have been very much confused. This is not at all surprising, for hardness and toughness are closely related. It would be well, therefore, to define these terms from the road maker's standpoint before going further.

HARDNESS.

There is no widely accepted measure of the property of hardness. Even in the case of metals, the hardness of which has received much study, there are many tests based on different conceptions of the term. The many methods devised for measuring hardness may be summarized under the following heads: Drawing substance under a point;[a] drawing a point over substance and determining resistance;[b] grooving with a standard edge and determining the depth of groove;[c] boring substance with a standard point and ascertaining depth of hole and number of revolutions;[d] grinding with a standard powder and taking the loss inversely;[e] compressing lenses on plates of substance and taking as the hardness the limit of pressure per unit of surface multiplied by the cube root of the radius of curvature;[f] compressing a point into substance and taking the volume of indentation inversely.[g]

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[a] Pekarek, Sitz. v. k. k. Akad. Wien, 1854, vol. 13. (Contains complete biography of earlier papers.)

[b] Turner, Proceedings Phil. Society, Birmingham, 1886.

[c] Pfaff, Sitz. k. k. Bayer. Akad., 1883.

[d] Pfaff, ibid., 1884.

[e] Rosiwal, Verhandl. k. k. Geol. Reichsanstalt, 1896, 17: 475.

[f] Auberbach, Wied. Ann., 1891–96.

[g] Report of Experiments on Metal for Cannon, United States Ordnance Department, 1856.

All of these tests were designed for substances of a homogeneous nature, and are consequently not at all suited to any of the road materials. Further than this, it can be seen that in their conception of hardness some of the investigators differ greatly. All of these methods, as well as modifications of them, are based either on abrasion or penetration.

The varying demands of technology give rise to different definitions and methods of testing, and the method used in any particular case must give a measure of the value of a new material for the purpose for which it is intended. To permanently deform a substance by compression with a pointed or spherical instrument tests altogether different properties from those opposed to abrasion.

Only one test has yet been devised for determining the hardness of road materials, and that, the writer believes, gives the value of this property as understood by road builders in a satisfactory manner. This is the Dorry test of the French School of Roads and Bridges which consists in grinding specimens with sand of a standard size and quality. This method of grinding with a powder has the advantage of having been used as a test for hardness by a number of the most able students of the subject, and at the same time valuable results have been obtained from it on the very class of materials in which we are most interested. Hardness, therefore, will be defined as the resistance which a material offers to the displacement of its particles by friction. The measure of hardness will be, inversely, as the loss of weight arising from the scoring by an abrasive agent.

TOUGHNESS.

In the consideration of road materials toughness is understood to mean the power possessed by a material to resist fracture under impact. As the surface of a road is continually subjected to the pounding of traffic, it can be seen that toughness is an important property from the standpoint of the road builder. From the laboratory standpoint the problem is not altogether a simple one, and considerable difficulty has been found in designing a suitable test for measuring the degree to which a road material possesses this property.

With homogeneous, structureless, brittle materials, resistance to impact may be due to a relatively low modulus of elasticity combined with high elastic limit. Provided a blow is delivered by a flat striking head with small local damage, on such a material toughness will be almost wholly due to elasticity. In this case there will be a critical energy of blow below which the specimen under test will not be broken by an indefinite number of blows, and in excess of which it will be broken by a single blow. The toughness of a road material in this instance will vary directly as the square of the elastic limit, which equals the ultimate strength, and inversely as the modulus of elasticity. In testing such materials under impact it would be necessary to apply a number of blows of successively increasing energy, and note that blow which causes failure. A test involving this principal will be described further on in detail.

With heterogeneous materials like most of the rocks used in road building, toughness depends on a number of factors. Among these may be mentioned interlocking of the crystals, the nature of the crystals themselves, and in some cases the nature of the cementing or binding agent. This matter has received but little study.

There is another class of road materials whose toughness is due to a combination of properties not considered under the above definition. It includes those materials capable of considerable deformation without rupture when stressed beyond the elastic limit. Asphaltum and tar practically constitute this class. They distinctly come under the head of tough substances; but it is obvious that toughness in this sense is viscosity and malleability.

CEMENTING OR BINDING POWER.

The binding power or, as it has now come to be called, the cementing value of a road material is the property possessed by rock dust or other finely divided material found in nature to act as a cement on the coarser fragments composing crushed stone or gravel roads. This property varies enormously, not only with different kinds of rocks, but also with those which are practically identical in classification and chemical composition. The absence of cementing power is so pronounced in some varieties of rock that they can never be made to compact with the road roller or under traffic. As the binder surface of a macadam or gravel road is most exposed to the action of wind and rain, as well as the wear and tear of traffic, it can be seen that the presence of this property is most essential to good results. Further than this, the hardness and toughness of the binder surface, more than of the rock itself, constitutes the hardness and toughness of the road, for if a load be sufficient to destroy the bond of cementation of the upper surface of a road, the stones below are soon loosened and forced out of place. The impervious shell obtained by the use of a rock of high cementing value gives the greatest protection to the foundation of a road. Moreover, it is a matter of common observation that a good surface which binds well is less dusty and less muddy, while the advantage from the standpoint of economy is very great, as it is only the loose, unbound material which is ordinarily carried away by wind and water.

In view, therefore, of the importance of this property, it has been made the subject of especial study in this laboratory. It was most important to know the cause of the cementing value in order to determine what could be done to improve the conditions of service. The results of this study, which have already been published as a contribution from the Bureau of Chemistry, have been very satisfactory.[a]

It appears that the cementing value depends upon a certain hydrated colloid condition of the particles, or some proportion of the particles. All rock powders that cement well are hydrated, i. e., contain water of combination, although it does not follow that all hydrated rock powders will cement. It seems that only a certain kind of water of combination is concerned with and measures the cementing value. This property is undoubtedly related to that of plasticity in clays, and, in a few words, is due to amorphous, inorganic particles which by reason of their characteristic porous structure are able to absorb and hold water, thereupon assuming a plastic and coherent condition. Heating above a certain temperature destroys this structure, and the powder no longer possesses the slightest cementing value. It is probable that this theoretical investigation will lead to important practical developments.

Under the head of physical tests will be found a description of the methods, as used in this laboratory, for testing the cementing power of road materials, in Table III, page 33, are given the maximum and minimum results obtained on various kinds of rock, and in Table A of the Appendix the individual results on each sample.

THE CAUSES OF THE DETERIORATION OF ROADS.

The agencies to be discussed as tending to the deterioration of roads include traffic (pounding of horses' feet, action of wheels, effect of load), weathering or chemical decomposition, frost, wind, and water, and may be classified as mechanical, chemical, and physical agencies.

MECHANICAL AGENCIES.

The severe pounding to which a road is subjected by the action of horses' feet tends to destroy the binding surface and loosen the underlying stones. The fine material when loosened is more readily carried off by wind or rain and water can then more readily penetrate to the foundation. The properties necessary to resist this action are, with stone blocks or brick pavements, high resistance to impact, and with crushed stone and gravel, binding or cementing power in the fine material of the surface and toughness in the larger fragments.

The effect produced on roads by the wheels of vehicles varies much with the material of which the road is composed and with the character of the traffic passing over it. If the surface of a road is perfectly smooth, and the load per running inch of tire is sufficiently within the limit of resistance of the surface, the amount of wear is probably insignificant. The moment, however, an irregularity of any kind occurs on the surface the wheels begin to pound at such points and water accumulates in the resulting depressions, which causes rapid deterioration. When the pressure per running inch of tire is too great for the road surface bad results soon follow. With crushed stone or gravel, not only should the pressure be within the limit of resistance of the rock of which the road is composed, but within the cohesive limit of the binder surface, for if this is not the case the passing wheels cause the material immediately beneath them to shift, furrowing the road and pressing the material up on either side of the furrow. This rutting is greatly increased by the consequent interference with the proper draining to the sides of the road, and the water is forced to accumulate on the surface, to gully out the road on grades, or make its way to the foundation. Always on a good road the resistance offered to compression by the material composing it exceeds the pressure of wheels, for if such were not the case the surface would soon be cut to pieces. It is, however, a frequent occurrence for the impact and even the pressure of wheels to exceed the limit of resistance of the binder surface of macadam and gravel roads. The pressure of wheels is distinctly beneficial to a road if it is sufficiently within the cohesive limit of the binding material and is evenly distributed over the surface. Indeed, it is necessary if a road is to be kept in a high state of perfection that it should be subjected to a sufficient amount of traffic to keep the material thoroughly compacted, and at the same time wear off a sufficient amount of fine material to take the place of that carried away by wind and rain.

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[a] On the Cause of the Cementing Value of Rock Powders and the Plasticity of Clays. Allerton S. Cushman, Jour. Am. Chem. Soc., May, 1903.

CHEMICAL AGENCIES.

It has frequently been remarked by writers on the subject, that the chemical decomposition of materials plays an important part in the deterioration of roads. If wood and asphalt are used this may be the case, but the writer is convinced that undue importance has been attached to this agency as regards most of the road materials. In a warm, dry climate there is practically no limit to the life of rock. This fact is exemplified by many of the structures of antiquity, such as the pyramids of Egypt, composed of limestone and granite, which have stood for many centuries, while the rock has undergone but little change. In such a climate this would be the case with almost any variety of rock, but in a humid climate with a great range of temperature less durability is to be expected. In all cases, however, the durability of rock is extremely great as compared with the life of a road. All rain and surface waters contain a sufficient amount of carbonic acid to dissolve carbonates of lime and iron, and when charged with the humus acids derived from the decomposition of animal and vegetable matter certain of the rock-making minerals containing magnesia, potash, soda, lime, iron, and even silica, are rendered soluble. There is also a small amount of sulphuric acid present in rain water in the vicinity of large cities. The rocks most affected by these reagents are the limestones and rocks having a matrix of lime and iron, but we find the resulting disintegration, in rocks likely to be used in road making, practically negligible when compared with that due to other agencies to which roads are subjected. Professor Pfaff, of Erlangen, subjected to atmospheric erosion specimens of limestone and granite. The annual loss undergone by the limestone was estimated to be equal to the removal of a uniform layer of the general surface about 0.01 mm in thickness, while the granite lost 0.0076 mm.[a]

Even with roads constructed of the most soluble rocks it can be seen that this action from a road-building standpoint is extremely slow. The crushed stone below the surface would be subjected to but little running water, while the amount of the binder that might be taken into solution would be insignificant when compared with the loss through other agencies of wear. The writer compared six thin sections of fresh diabase (trap) with six other sections from the same rock ledge which had been in a roadbed for nine years, and so far as could be observed with the microscope no change whatever had taken place in the plagioclase and augite of which the rock is composed, or in the rock as a whole. In specimens of trachyte examined in the same way, one of which had been in a roadbed for twelve years, no alteration was noticeable. This subject has been fully treated by the writer in a former publication.[b]

PHYSICAL AGENCIES.

It is very doubtful whether the effect of freezing is injurious to road materials that absorb water even in considerable amounts, but at all events it is probably very small when compared with the destruction wrought by this same agency on the roadbed as a whole. One hundred volumes of water expand on freezing to 109 volumes of ice. If expansion is prevented the pressure developed on cooling the water 1 degree below its normal freezing point is 144 tons per square foot. When we examine the subject closely it becomes apparent that the action of frost on road materials is not so great as it at first seems. In the following table is given the percentage of water absorbed by the more important road materials.

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[a] Pfaff, Sitz. k. k. Bayer. Akad., 1888.

[b] Report of the Massachusetts Highway Commission, 1900.

TABLE I.—*Water absorbed by the principal rock species.*

Name of material.	Number of samples.	Maximum.	Minimum.	Name of material.	Number of samples.	Maximum.	Minimum.
		Per cent.	*Per cent.*			*Per cent.*	*Per cent.*
Amphibolite	6	0.32	0.10	Granite	15	0.89	0.06
Andesite	3	1.30	1.16	Limestone	46	2.84	.03
Basalt	6	4.34	.06	Peridotite	1	.14	.14
Chert	11	2.72	.25	Quartzite	4	.82	.06
Conglomerate	2	2.43	1.60	Rhyolite	9	2.40	.02
Diabase	12	.48	.05	Sandstone	7	8.84	.30
Diorite	7	.59	.11	Schist	16	.59	.11
Dolomite	16	2.95	.04	Shale	8	3.07	.81
Eclogite	2	.04	.03	Slag	2	2.57	.34
Felsite	2	.20	.01	Slate	2	.30	.24
Flint	1	.13	.13	Steatite	1	.11	.11
Gabbro	1	.05	.05	Syenite	1	.18	.18
Gneiss	6	.18	.05				

The table shows that the amount of water absorbed by the best rocks for road making is small, but even where the amount is very large the action of frost does not seem to be markedly injurious. The following statement has been made concerning the effects of frost on paving brick:

> The last two years the city of Peoria has conducted freezing tests, which are not completed, but they have gone far enough to show that the average brick could absorb 6 or 8 per cent and still not be affected by repeated freezing. If the absorption is that high, the brick would be soft and the wearing away under traffic would be such that the brick should be rejected for paving.[a]

While subjecting saturated paving bricks or fragments of rock to a freezing temperature may injure them but little, yet when an entire road surface freezes the effect is very different, for the absorptive power of the road is, with most materials, far greater than that of the material itself, and the strength of the road as a whole is far less than that of its individual components. In the case of pavements which have but little yield, such as brick and stone block on concrete foundations, the stresses set up by freezing must be very great.

By far the most destructive work of frost is effected on crushed stone or gravel roads. If the construction is defective and the maintenance poor, water accumulates in the body of the road, which spreads the material so much on freezing that the bond in some cases is destroyed, and in other cases much weakened. Experiments have been made to determine the increase in volume of wet rock dust, similar to that composing the binder of macadam roads, when subjected to the action of frost. Compressed briquettes of the dust were saturated with water and exposed to a temperature considerably below the freezing point for twelve hours. They were carefully measured with a micrometer screw before and after freezing and were found, on the average, to have increased in volume by about one-half of 1 per cent, the maximum increase being less than 1 per cent.

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[a] Proceedings of the Illinois Society of Engineers and Surveyors, 1897.

The agencies of destruction to roads, which have already been considered, are alike in one respect—they cause only abrasion and disintegration to the materials composing the road, while wind and rain carry away all the material loosened by these factors. Wind and rain are, therefore, the chief agents that remove material from a road, and as their action is incessant the loss is very great. From elaborate measurements made by the National School of Roads and Bridges of France, it appears that about 7 cubic yards of material per mile per annum are transported from the macadam roads of France. Undoubtedly this loss must vary widely in different localities and with different materials, and therefore such general results are not applicable to individual cases. The accumulation of water on the surface and its constant penetration to the foundation, together with the continual washing that takes place, make the action of water the most serious element of destruction that the road builder has to combat.

SERVICE TESTS.

The term "service test," as here used, does not refer to the informal tests that every material receives in service, the results of which form the main part of our experience with it, but to those formal, competitive tests that are frequently inaugurated in order to determine the relative merit of various articles or the efficiency of various processes. In the case of paving bricks, for instance, a number of different brands are laid in the same street and subjected to the action of traffic for an extended period, observations of the rate of wear being made from time to time.

These service tests in the case of road materials are of limited value. In the first place, it is difficult to determine the exact significance of the observed results because of the variability of the conditions and the indefiniteness of our knowledge of them. Further, the results of the test can not always be expressed definitely or in units that will be agreed upon, and thus much confusion will arise in the attempt to report the results. Again, a given material which proved best under one set of conditions with respect to climate, traffic, and topography, might be entirely unsuited to different conditions. Another practical objection is that a large expenditure of money and a prohibitory lapse of time, involving in some cases a change of traffic conditions, are necessary before any conclusions can be drawn relative to a matter which usually must be settled without great expense or delay.

The value of materials used for other purposes than road construction is in most cases satisfactorily determined by laboratory tests in which the exact conditions of service may or may not be approximated, according as it is difficult or impracticable to duplicate them. The essential requirement of such laboratory tests is that they shall be uniform, well defined, and directed to the measurement of the useful properties of the material. The samples must be submitted to the conditions which are exactly known and the elements or combination of elements of endurance and value isolated and determined quantitatively.

The nature of the agencies of service being known, and the degree to which the material possesses mechanical properties useful in resisting these agencies having been determined by test, the value of the material for any given service may be predicted. In case materials do not differ widely in some element of value it is necessary to resort to known and constant conditions of laboratory tests in order to determine their relative merit. The reliability of laboratory tests may be determined by applying them to appropriate materials the quality of which is known from experience in service. It is well to remember that what are apparently small differences of quality in a material will result in large differences of expense of maintenance and of road life. It is not too much to say that the life of a macadam road costing from $4,000 to $10,000 per mile may be from two to twenty years, depending upon a proper choice of material to suit the local conditions. The need and usefulness of proper laboratory tests to determine the essential qualities of road materials is thus evident.

Certain formal competitive tests of materials are, however, to be noted. Various brands of paving brick have been laid in the same street with a view to determining their relative merit and to check the results of the rattler test. Such tests are in progress in the cities of Washington, Baltimore, and Detroit.

In France elaborate data have been collected. In 1865 tests were begun on the national roads of France to determine the quality of the various materials of construction and ultimately to ascertain for each department the materials best adapted for its roads. Careful measurements were taken of the depth of stone for all the national roads by digging trenches, alternately from one side of the road to the center, so as to give a complete section of one-half of the road. This test was repeated in 1874, 1886, and 1893, the last measurements requiring over a half million trenches. These measurements were taken for the purpose of determining accurately the amount of stone worn off annually. At the same time a census was taken of the traffic passing over each road. Each division engineer was also required to build short sections of road of each available rock in his district along the same line of travel. All the materials of construction and maintenance were carefully measured and the results of wear observed.

Observations were taken over 22,000 miles of road. The traffic was classified and rated as follows:

(1) Each horse hauling a public vehicle or a cart loaded with produce or merchandise, 1.

(2) Each horse hauling an empty cart or a private carriage, $\frac{1}{2}$.

(3) Each horse, cow, or ox unharnessed, and each saddle horse, $\frac{1}{5}$.

(4) Each small animal (sheep or goat), $\frac{1}{20}$.

A record of traffic was made every thirteenth day throughout the year, and an average taken to determine the amount. The general average over all the roads in 1893 was 170.6 units of travel. To repair the roads there had been required an average of 49 cubic yards per mile and per 100 units of travel. The detailed accounts of this work are published in the official report of the minister of public works of France,[a] and offer a mass of data which, when combined with the results of laboratory tests, should be of great service in determining the value of the latter. The results of these elaborate service tests show, in general, a decided agreement with the results of the laboratory tests which were conducted at the same time. Since the investigation described laboratory tests have been the basis of selection of road materials in France.

In Portugal some attempt has been made to determine on a large scale the wear of roads and its relation to laboratory tests. A census of the traffic was kept at the tollgates and the wear was determined by making observations of the thickness of the roadbed whenever excavations had to be made. The results of these observations have not yet been published, but in a ministerial report it is stated that the laboratory tests indicated very clearly the best materials for use.

HISTORICAL REVIEW OF ROAD-MATERIAL TESTS.

It is only comparatively of recent years that tests of materials of construction have been carried on in a systematic way and the history of road-material testing is of still more recent date. Doubtless the ancient Roman engineer satisfied himself of the suitability of his materials to the end in view, but this was probably done in a very general way, or some records would have come down to us of the methods employed. Many of the earlier writers on macadam road building noted the superiority of wear in certain varieties of rock, and reference is often made to the desirability of hard and tough rock. As early as the middle of the last century compression tests were made on rocks in the endeavor to determine their road-building quality. The systematic testing of road materials may be said, however, to have been begun during the decade opening with 1870, in France, where it has been steadily developed ever since. The Portuguese Government, the next to take up the subject, adopted some of the French tests and conducted them with much precision. While the importance of the subject has been recognized in England, yet aside from the limited investigations of a few individuals, almost nothing has been done. The same is true, even to a greater extent, of Germany, and the other continental nations have left the subject practically untouched. The United States is the only other country where this work has been carried on in a systematic way.

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[a] Campau, A. P. Rockwell, Roads and Pavements in France.

In 1893 the Massachusetts highway commission, in collaboration with the Lawrence Scientific School of Harvard University, established a road-material laboratory at the latter institution. Although paving-brick tests had been made previously, this was the first laboratory in the United States for testing road materials in a systematic way. The Deval abrasion test was adopted, and tests for determining the cementing power of rock dust were first developed there. Since then laboratories have been equipped with appliances for testing road materials by the Maryland Geological Survey, Columbia University, Wisconsin Geological Survey, Cornell University, the University of California, and the United States Department of Agriculture.

TESTS OF ROAD MATERIALS IN FRANCE.

In December, 1878, the French commission on national roads decided to introduce certain mechanical tests at the laboratory of the School of Roads and Bridges, to be conducted in addition to and parallel with service road tests. The Deval test, bearing the name of its designer, for determining the resistance offered by macadam rocks to abrasion had already been pronounced by the street department of the city of Paris to be useful and reliable in testing the rock used in contract work and for selecting new quarries. This test was accordingly adopted by the commission and a laboratory was founded, which has steadily increased in usefulness.

The Deval Machine.

This machine, as far as the writer has been able to ascertain, was the first one designed especially for testing road materials. It was first exhibited at the Paris Exposition of 1878, and then consisted of two iron cylinders fastened to a shaft, so that the axis of each cylinder was at an angle of 30 degrees with the axis of rotation. The shaft which held the cylinders was supported on bearings, and had at one end a pulley wheel by which the cylinders were revolved and at the other a revolution counter. Each cylinder, 20 cm in diameter and 34 cm in depth, was closed at one end and had a tightly fitting iron cover for the other. A cut of a similar machine slightly modified and in use in the Road Material Laboratory is shown in fig. 1.

In adopting the Deval machine, the only change made by the School of Roads and Bridges was to increase the number of cylinders to eight in order to increase the output. The eight cylinders are mounted four by four on two parallel shafts geared to rotate with the same rapidity. This arrangement renders it possible to make eight tests simultaneously. The method of operation is as follows:

The rock to be tested is broken in sizes as nearly uniform as possible, each fragment being made to pass in all positions through a 6-cm ring. Five kilograms of rock thus prepared and previously cleansed by washing and subsequent drying are placed in one of the cylinders. The cover is then bolted on and the cylinders revolved at the rate of 2,000 revolutions per hour. The fragments of stone are thrown from one end of the cylinder to the other twice in each revolution, and thus grind and pound against one another and the ends of the cylinder. At the end of five hours, or 10,000 revolutions, the machine is stopped, the cylinders opened, and the contents emptied into a basin. The cylinder and the cover are carefully washed and the water used is poured into the same receptacle. Each stone is then washed and brushed under the water until all adhering dust is removed. After drying, the detritus is separated into the following three sizes: Above 1 cm, between 1 cm and 0.16 cm, and below 0.16 cm. Only the

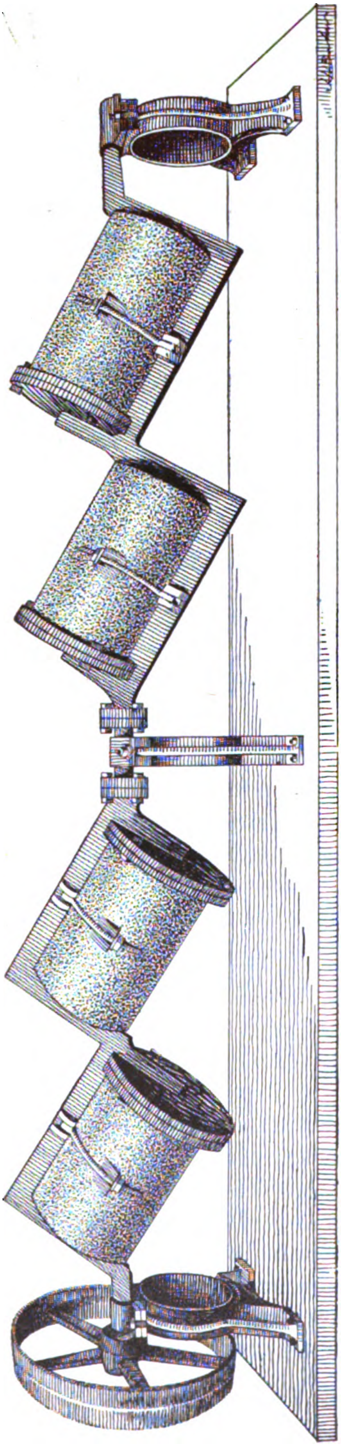

FIG. 1.—Abrasion machine.

material below 0.16 cm, the weight of which is recorded, is used for the purpose of the test.[a]

At first a standard rock of superior wearing quality was always placed in one of the cylinders as a standard of comparison, and the proportion of the weights of the dust under 0.16 cm from the standard rock and from the rock to be tested was assumed to give their relative resistance to abrasion. It was found, however, that only the best varieties of rock gave less than 100 grams of powder under 0.16 cm, i. e., 20 grams per kilogram of rock, or 2 per cent of their weight. The number 20 was therefore adopted as a standard of excellence, and the "coefficient of wear" for any rock tested may be obtained by the following formula:

$$\text{Coefficient of wear} = 20 \times \frac{20}{W} = \frac{400}{W}$$

in which "W" is the weight in grams of the detritus under 0.16 cm in size obtained per kilogram of rock used.[a]

COMPRESSION TEST.

In addition to the test made with the Deval machine the following compression test was later adopted: This test is made on 25 mm cubes of rock with a hydraulic compression machine. The cubes are sawed at the laboratory from specimens carefully selected to represent the average quality of the rock. At least three cubes of each sample are tested after desiccation either in the open air or at a temperature of 40° C., or after being saturated with water. The resistance offered by each cube is obtained in kilograms per square centimeter of bearing surface, and the average of the results furnished by the different cubes is used.

Experience having shown that the hardest rocks rarely resist a load greater than 2,000 kg per square-centimeter section, the commission adopted the coefficient 20 for the materials having this degree of resistance. This corresponds approximately to the resistance to compression of wrought iron. The coefficient of any rock, therefore, may be obtained by the following formula:

$$\text{Coefficient} = \frac{3{,}000}{20} + E = 150\ E.$$

"E" represents the breaking load in kilograms per square-centimeter section. To determine the density of rock the cubes used for the compression test are measured and weighed. This is undoubtedly the most accurate of any of the simple methods for making this determination.

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[a] Results obtained from this test in the Road Material Laboratory are given in Table II, page 26, and in Table A of the Appendix.

THE DORRY TEST.

The test for hardness is made with the Dorry machine. The specimens to be tested are sawed into rectangular prisms 8 cm high with a base 4 cm by 6 cm. These specimens are placed, two at a time, so that they rest on the upper surface of a circular grinding disk of cast iron, which is rotated in a horizontal plane by a crank. They are held in clamps so arranged that the bases of the specimens rest on alternate sides of the grinding disk 26 cm from the center. The specimens are weighted so that they press against the grinding disk with a pressure of 250 grams per square centimeter. Sand of a standard quality and size, obtained by crushing quartzite rock and screening it, is fed onto the disk from a funnel. The quantity of sand used in each test is 4 liters. The disk is rotated at the rate of 2,000 revolutions per hour for two hours. A drawing of this machine, slightly modified, is reproduced in fig. 2.

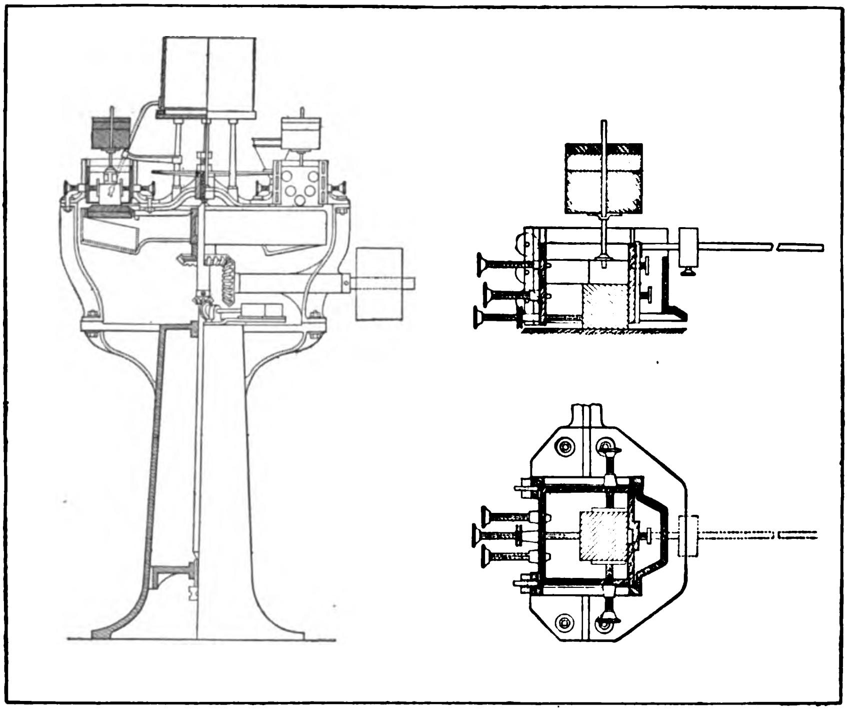

FIG. 2.—Dorry machine for determining hardness.

After 2,000 revolutions the specimens are reversed to ascertain if there is any difference in wear between the two ends and to make the result approach more nearly a general average of the samples. The diminution in the height of the specimen is measured and its loss in weight determined after each 1,000 turns of the grinding disk. No coefficient of wear has been established for this test, but the loss in height undergone by each specimen after 4,000 revolutions of the disk is taken as the result of the test and serves for comparison. Tests are always made on at least three specimens of each sample, and the final result is taken from their average.

IMPACT TEST.

The impact test is made with a machine especially designed for the purpose at the national laboratory. It resembles a pile driver very closely in principle, consisting of a hammer with vertical guides to direct its fall. The hammer is raised by a cord which passes over a pulley at the top of the guides, and can be released at any desired height, from which it falls upon the test piece which is held below by clamps. Two hammers are employed, one weighing 42 kg and the other 20 kg with falls of respectively 100 cm and 80 cm. The number of blows necessary to crack the specimen, and also the number necessary to produce its complete destruction, is noted. The test is made upon 4 cm cubes, at least three cubes being used for each specimen with each hammer. Occasionally some other tests are used, including the determination of porosity, the effect of frost, and transverse breaking.

TESTS ON WOOD PAVING BLOCKS.

The tests most commonly used for wood paving blocks consist of the determination of their resistance to wear when saturated with water; resistance to compression, resistance to impact, the determination of expansion by absorption of water, and the measurement of the thrust exerted when expansion is partially prevented. The test for wear is made with the Dorry machine. The specimens consist of prisms of the same dimensions as those used for testing the hardness of paving stones. The specimens are placed in the machine with the grain of the wood at right angles to the grinding disk. The only difference in the conduct of the test is that emery is used as the grinding agent instead of quartz sand. Only the loss in height of the specimen is recorded.

The compression test is made with a hydraulic press upon prismatic specimens, with a base 8 cm square and a height equal to that of a paving block, the grain of the wood being parallel to the direction of the load. The test specimens are either dried at a temperature between 30° and 40° C., or they are saturated with an amount of water estimated to be equal to that absorbed by a paving block in service.

The resistance to impact is tested with the machine described above upon prisms having a base of 6 cm square and a height equal to that of a paving block. The test piece is placed in a cast-iron case 7 cm square, open both at the top and the bottom, and is held in place by a resinous cement which completely surrounds it, the top of the prism projecting about 1 cm above the case. The 20 kg hammer is employed in this test, with a height of fall of 200 cm. The number of blows of the hammer which cause an appreciable breakage is noted. Failure in the test piece is indicated by a smaller rebound of the hammer and a diminution in the height of the specimen. Three dried and three wet specimens are subjected to this test.

For determining the thrust caused by absorption when expansion is partially prevented, whole paving blocks are used. The block, after thorough desiccation, is placed in a water-tight receptacle and held between two plates at top and bottom, so that the top face of the test piece rests against a block of cast iron, which is stationary. The lower plate is supported on the small lever arm of a cement-testing machine. Water at a temperature of 30° C. is poured around the paving block, and as expansion takes place the lever arm of the machine tends to rise. When it rises a valve is opened through which mercury pours into a vessel supported at the end of the arm till the arm comes back to mid-position, when the valve is closed. As the lower plate is fixed when the long lever is in its mid-position, and as this arm is always kept automatically in that place the specimen is prevented from extending longitudinally. The weight of the mercury in the vessel at the end of the short arm is at any moment a measure of the force exerted by the test piece, and as the vessel is supported by a spring balance, this can be read at any, time. Observations are made at first at intervals of three hours then at intervals of twelve hours, and finally at intervals of twenty-four hours till all increase ceases. It will be seen that while this test does not reproduce closely the conditions that exist in practice, the results are of value in giving some indication of the amount of thrust to be expected. After the test the specimens are weighed to ascertain how much water has been absorbed.

No mechanical tests have yet been devised, or considered necessary, for asphalts. Chemical analyses are made, however, to determine the proportion of bitumen, sand, calcium carbonate, clay, pyrites, etc.

TESTS OF ROAD MATERIALS IN PORTUGAL.[a]

The laboratory testing of road materials was first taken up in Portugal in 1881, in the district of Santarem. The Deval test of the French School of Roads and Bridges was adopted, and over sixty varieties of rock were tested. The basalts, diorites, and certain of the granites from the valley of the Tagus gave coefficients of wear as high as 24. This is a higher coefficient than that of flint, which previous to this test was thought to be the best macadam rock, but subsequent trials on the road have proved this to be a mistake. The coefficients of the limestones tested ranged from 2 to 17. Those from

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[a] Jornal dos Engenheiros Civiles Portuguezes, 1882, 13: 150. Revista das Obras Publicas e Minas, vols. 13 and 24; also special ministerial report.

10 to 17 gave good results on roads where travel was light, while those below 10 were found to be too soft.

Compression tests are also carried on at the laboratory for testing materials at Lisbon, which is equipped with three compression machines. Tests can be made on 10 cm, 7cm, and 5 cm cubes, as desired but the smaller cubes, which are crushed both dry and wet, are generally used for testing road materials. No coefficient has been worked out for this test. The number of kilograms per square centimeter required to destroy a test piece is used for comparison. All samples are sent to the laboratory by the directing engineer of the district from which they come. The results of these laboratory tests have been found to be in agreement with the limited observations made on the actual wear of roads.

HISTORY OF PAVING BRICK TESTS.

An account of the work of the National Brick Manufacturers' Association in establishing a standard rattler test, and of the critical and confirmatory studies of other investigators arising therefrom, would give a fairly complete history of the development of paving brick tests.

Formerly reliance was placed entirely on the decisions of the street inspector, who rejected individual bricks that were defective in form or hardness. The necessity for such work has not disappeared, even with the development of the present perfected tests. Many engineers have used for some time the cross breaking and crushing tests, or else the absorption test as an index of internal structure, but gradually specific tests and standards of quality have been reached through experience.

The rattler test has been used for some years. In 1895 the National Brick Manufacturers' Association appointed a commission on paving brick tests. The first report[a] of work, issued in 1897, included the results of the following studies: The rattler test by Edward Orton; the absorption test by H. G. Wheeler; the cross-breaking test and additional studies of the rattler test by F. F. Harrington, and specifications for the cross-breaking test by J. B. Johnson. As a consequence of these reports the rattler test was chosen as the most significant and reliable, and the size and speed of the rattler and the duration of the test were specified in accordance with the present standard, although no abrasive shot were then used in the rattler. The absorption test was condemned because it did not indicate the the relative value of different brands of paving brick. It was admitted, however, that there existed in the case of any given brick a particular per cent of absorption which indicated whether the brick had been properly burned. The fact was also brought out that an observation extending over a period of less than one week was not of sufficient duration to represent the entire absorptiveness of the brick. The directions given for conducting the test were to weigh five abraded bricks after drying for forty-eight hours at a temperature of 250° F., then immerse them for forty-eight hours and reweigh.

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[a] Report of the Commission appointed to Investigate the Subject of Paving Brick Tests. T. A. Randall & Co., Indianapolis.

The cross breaking and compression tests were left optional. For the former test knife-edges rounded both transversely and longitudinally were recommended and ten bricks were to be tested. For the latter test five half bricks were to be used and loaded on edge through plaster of Paris beds.

About the same time a study of the rattler test was conducted at the University of Illinois by A. N. Talbot to determine the proper composition of an abrasive agent for use in the rattler test. It appeared[a] that it was necessary to use a mixture of large and small cast-iron shot in order to distinguish between soft and hard brick in the rattler test.

Minor studies of the rattler test were made at Purdue University by W. K. Hatt[b] and at the State Agriculture College of Iowa by A. Marston.[c] The former showed that the moisture condition of the brick was important. The latter made a study of the cross-breaking test and showed that the large variation in individual results was due to real differences in the quality of the bricks tested.

Gomer Jones[d] described a rattler in which the bricks were held in pockets cast on the staves so that the interior of the rattler was lined with brick, on the surface of which lining the shot filling rolled and produced impact and abrasion. The committee on technical investigations of the National Brick Manufacturers' Association subjected Talbot's "shot process" and the Gomer-Jones process to a thorough examination by comparing the action of the two processes on bricks of known grade. The results reported in 1900[e] favored the use of that form of shot suggested by Talbot, and a new standard of the National Brick Manufacturers' Association, which prescribed a shot filling, was accordingly adopted. The Jones rattler was found to be mechanically defective. This rattler was improved by Talbot and a new investigation of the action of the improved rattler compared with the action of the standard process was instituted. The results given in a preliminary report of the committee of the National Brick Manufacturers' Association in August, 1901, showed that reliance could be placed on both the standard process and the Gomer-Jones process as modified by Talbot. A description of the standard method as finally adopted by the National Brick Manufacturers' Association will be given among the methods of the Road Material Laboratory.

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[a] The Technograph, University of Illinois, No. 12, 1898.

[b] Proceedings Indiana Engineering Society, 1899.

[c] Proceedings Iowa Engineering Society, 1899.

[d] Columbus Meeting of N. B. M. A., 1899.

[e] Detroit Convention, February, 1900.

METHODS AND TESTS OF THE ROAD MATERIAL LABORATORY.

The Road Material Laboratory was established in December, 1900, in the Bureau of Chemistry. Up to the present time reports have been made on about five hundred samples, representing a geographical distribution over thirty-eight States of the Union and including a number of samples from Porto Rico and Cuba.

The primary object of this laboratory is to make standard tests on road materials, free of charge, for citizens of the United States. In addition to this allied problems may be presented for study, such as the suitability of clays for the manufacture of paving bricks, drain tiles, cements, etc., the testing of cements and concretes for road foundations, drains, gutters, and highway bridges. It is the intention of the Department to aid as far as possible in the solution of all the problems of road building, but more particularly those relating to rural highways. It is not, however, the policy of the Department to undertake scientific investigation or tests of materials for manufacturers or others who desire to use the information thus acquired to promote commercial ends.

Any person desiring to have road materials tested will, on application, be supplied with instructions for collecting and shipping samples to this laboratory. The tests as carried on at present are described in the following pages.

TESTS FOR ROCK AND GRAVEL.

ABRASION TEST.

This test is almost identical with the Deval test already described under the French methods, the only difference being the omission of certain features not deemed strictly necessary, and a few modifications in the machine to simplify its operation and lessen the cost of construction. This machine, shown in fig. 1, p. 18, is made entirely of cast iron and has given excellent service. The test is made in the following manner: The sample to be tested is first broken in pieces that will pass in all positions through a 6 cm (2.4-inch) ring, but not through a 3 cm (1.2-inch) ring. The stones are then cleansed, dried in a hot-air bath at 100° C., and cooled in a desiccator. Five kilograms are weighed and placed in one of the cylinders, the cover bolted on, and the machine rotated at the rate of 2,000 revolutions per hour for five hours. When the 10,000 revolutions of the machine are completed the contents of the cylinder are placed on a sieve of 0.16 cm ($\frac{1}{16}$ inch) mesh, and the material which passes through is again sifted through a sieve of 0.025 cm (0.01 inch) mesh. Both sieves and the fragments of rock remaining on them are held under running water till all adhering dust is washed off. After the fragments have been dried in a hot-air bath at 100° C. and cooled in a desiccator they are weighed and their weight subtracted from the original 5 kg (11 pounds). The difference obtained is the weight of detritus under 0.16 cm ($\frac{1}{16}$ inch) worn off in the test, from which the French coefficient of wear is determined by the formula already given. Besides the French coefficient, the percentage of material under 0.16 cm in size is also reported. For a time another coefficient, called the Department coefficient, was used, which was obtained by subtracting 4,000 grams from the weight of the remaining fragments over 3 cm (1.2 inches) in size and dividing the difference by 10. This allows a possible range in results from 0 to 100, i. e., if 1,000 grams or 20 per cent of the material is abraded from the original 5 kg, the result will be 0, and the material is considered unfit for road making; if no dust is worn from the original 5 kg the result will be 100.

Although both the French and the Department coefficients are arbitrary, they have a distinct value. The French coefficient has been in use for many years, and is familiar to road builders throughout the East. At present both the French coefficient and the per cent of wear are reported, and the Department coefficient will also be given on application. In Table II are given the maximum and minimum percentages of wear and the French coefficient for all varieties of rock that have been tested in this laboratory, and in Table A of the Appendix the results on each sample tested are given.

TABLE II.—*Abrasion test.*

Name of material.	Number of samples.	Per cent of wear.		Coefficient of wear.	
		Maximum.	Minimum.	Maximum.	Minimum.
Amphibolite	6	4.6	1.5	26.7	8.5
Andesite	3	6.4	2.6	15.2	6.3
Basalt	6	16.6	1.7	23.6	2.4
Chert	10	27.9	2.7	14.6	1.4
Clay	1	19.1	19.1	2.1	2.1
Conglomerate	2	12.7	5.7	7.0	3.2
Diabase	14	6.3	1.2	34.5	6.4
Diorite	8	3.4	1.9	20.6	11.7
Dolomite	15	18.6	2.5	16.1	2.2
Eclogite	1	2.9	2.9	13.8	13.8
Felsite	2	3.4	1.9	21.3	11.9
Field stone (erratics)	1	5.0	5.0	8.0	8.0
Flint	1	7.2	7.2	5.5	5.5
Gabbro	1	2.9	2.9	14.3	14.3
Gneiss	6	5.2	1.9	20.7	7.7
Granite	15	14.8	1.8	21.7	2.7
Limestone	46	34.2	2.8	14.2	1.2
Mixed stone	1	8.7	8.7	4.6	4.6
Peridotite	1	3.6	3.6	11.1	11.1
Quartzite	3	3.6	2.2	18.2	11.1
Rhyolite	9	9.7	1.8	22.9	4.1
Sandstone	7	31.8	3.4	11.6	1.3
Schist	16	7.1	1.9	20.6	5.7
Shale	2	16.2	4.7	8.6	2.5
Slag	2	8.3	7.6	5.2	4.8
Slate	2	6.9	4.7	8.5	5.8
Syenite	1	2.8	2.8	14.4	14.4

Cementation Test.[a]

The binding or cementing power of rock dust is such an important element in road building that much time has been spent in the endeavor to devise a suitable test for determining the degree to which the various rocks and gravels possess this property. Many tests have been tried, but as yet only an impact test, carried on in a uniform manner as described below, has given satisfactory results.

One kilogram of the rock to be tested is broken sufficiently small to pass a 6 mm but not a 1 mm mesh screen. It is then placed in a ball mill and is ground for two hours and a half. This ball mill, shown in fig. 3, contains two chilled iron balls which weigh 25 pounds each, and is revolved at the rate of 2,000 revolutions per hour. It was found by experiment that grinding rock thus prepared for two hours and a half was sufficient to reduce it to a powder that would pass through a 0.25 mm mesh. The dust thus obtained is mixed with water to about the consistency of a stiff dough, and is kept in a closed

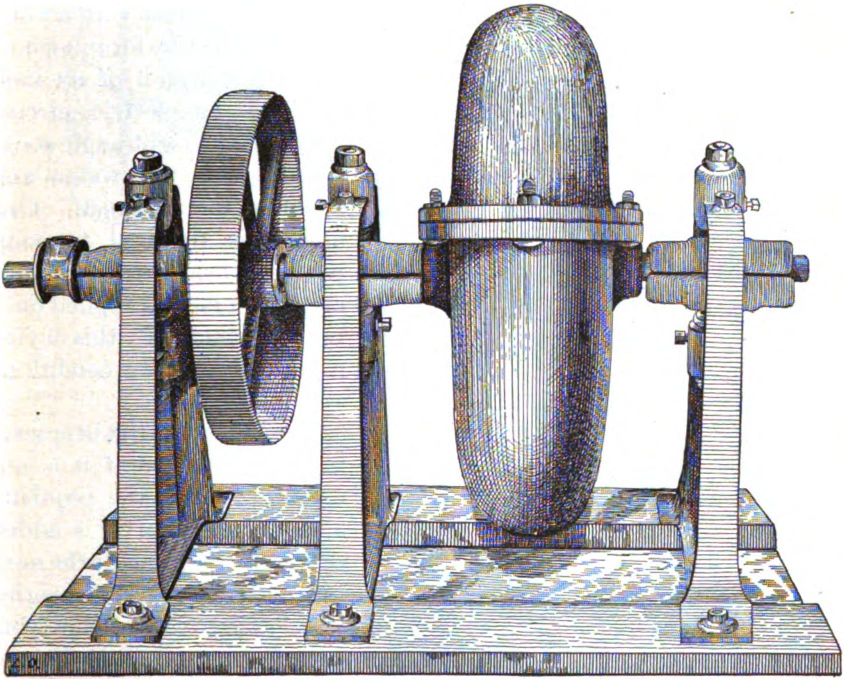

FIG. 3.—Ball mill.

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[a] This test, and the necessary machines for conducting it, were designed and developed by the writer for the Massachusetts Highway Commission and the Road Material Laboratory. The impact machine at present used was built especially for this laboratory by the Maryland Geological Survey, under the direction of Mr. A. N. Johnson, highway engineer to the survey, who made several useful modifications in the machines.

jar for twenty-four hours. About 25 grams of this dough is placed in a cylindrical metal die, 25 mm in diameter, which can be seen in fig. 4. A closely fitting plug, supported by guide rods, is inserted over the material, which is then subjected to a pressure of 100 kg per square centimeter.

It is most important that these briquettes should be compressed in a uniform manner, and for this a special machine has been designed (fig. 5). The die is placed on an iron platform supported by a piston rod, which is connected directly with a hydraulic piston below. Water from a tank is admitted to the hydraulic cylinder through a small orifice in the pipe. As the piston rises the platform and die are carried up with it, the plug of the latter coming in contact with a yoke attached to a properly weighted lever arm. When the lever arm is raised one-eighth of an inch it closes an electric circuit which trips a right-angle cock, shutting off the water and opening the exhaust. One minute is required to compress a briquette, and the maximum load is applied only for an instant. By this device practically uniform conditions are obtained.

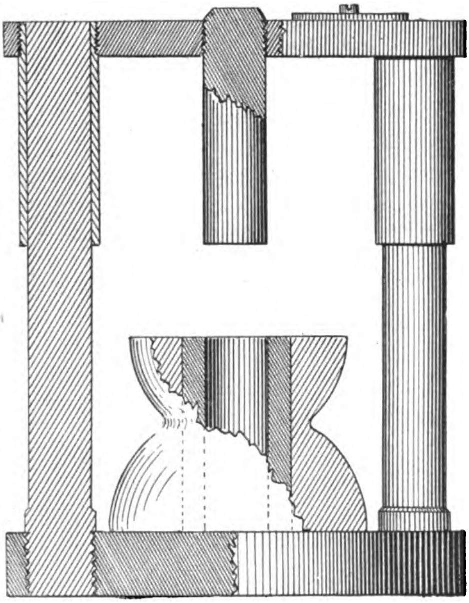


FIG. 4.—Briquette die.

The height of the briquette is measured, and if it is not exactly 25 mm the requisite amount of material is added or subtracted to make the next briquette the required height. Five briquettes are made from each test sample, and allowed to dry twelve hours in air and twelve hours in a steam bath. After cooling in a desiccator they are tested by impact in a machine especially designed for the purpose (fig. 6). It consists of a 1 kg (2.2 pounds) hammer (*H*), which is guided by two vertical rods (*D*). The hammer (*H*), which ends in a small cone at the top (*L*), is caught on the lower side of the cone by two spring bolts (*S*) and is lifted by a crosshead (*I*) which is joined to a crank shaft above. A vertical rod (*P*), which is directly over the hammer cone, can be adjusted by thumbscrews to give a drop to the hammer varying from a fraction of a millimeter to 10 cm. This rod has a hollow cone at its lower end into which the cone of the hammer head is thrust when the hammer is lifted by the crosshead (*I*). When the cone of the hammer head is brought into the cone of the adjusting rod the hammer is

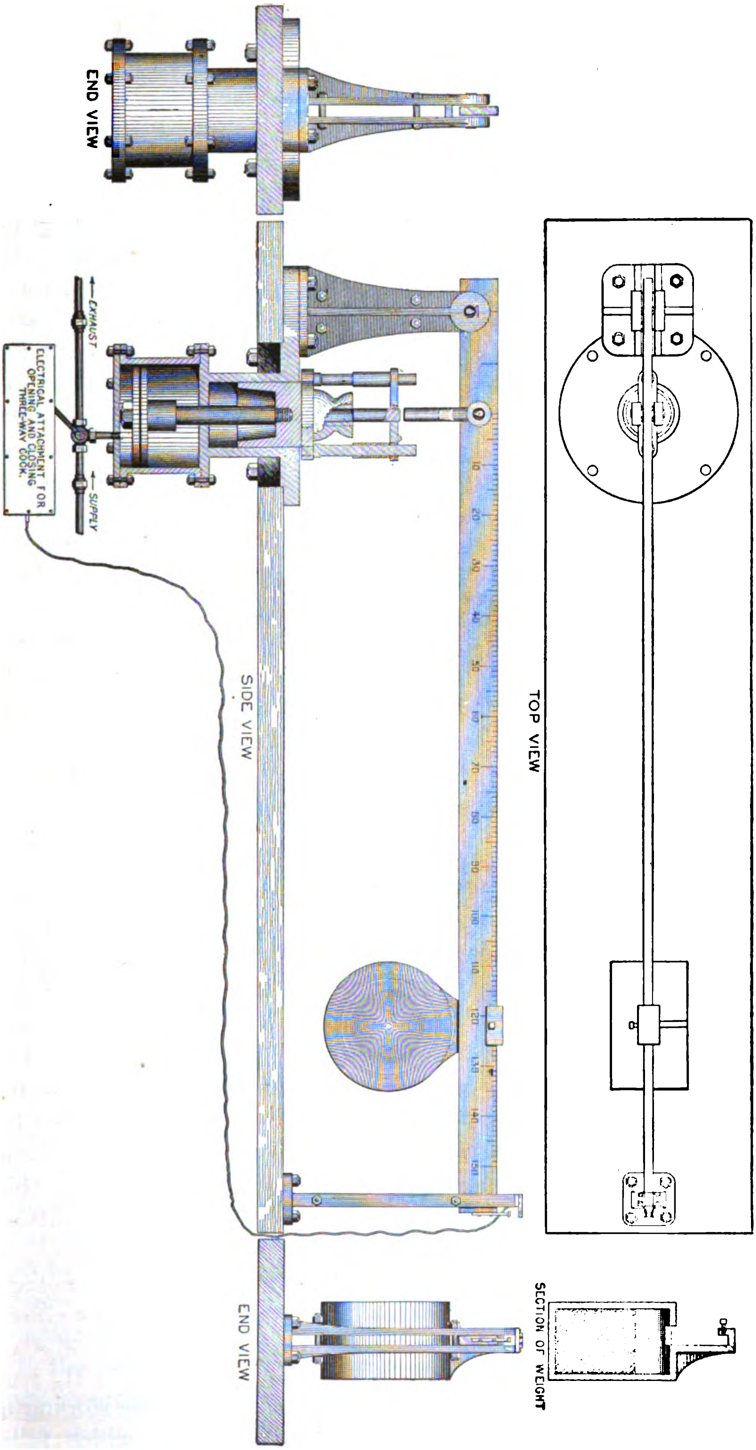
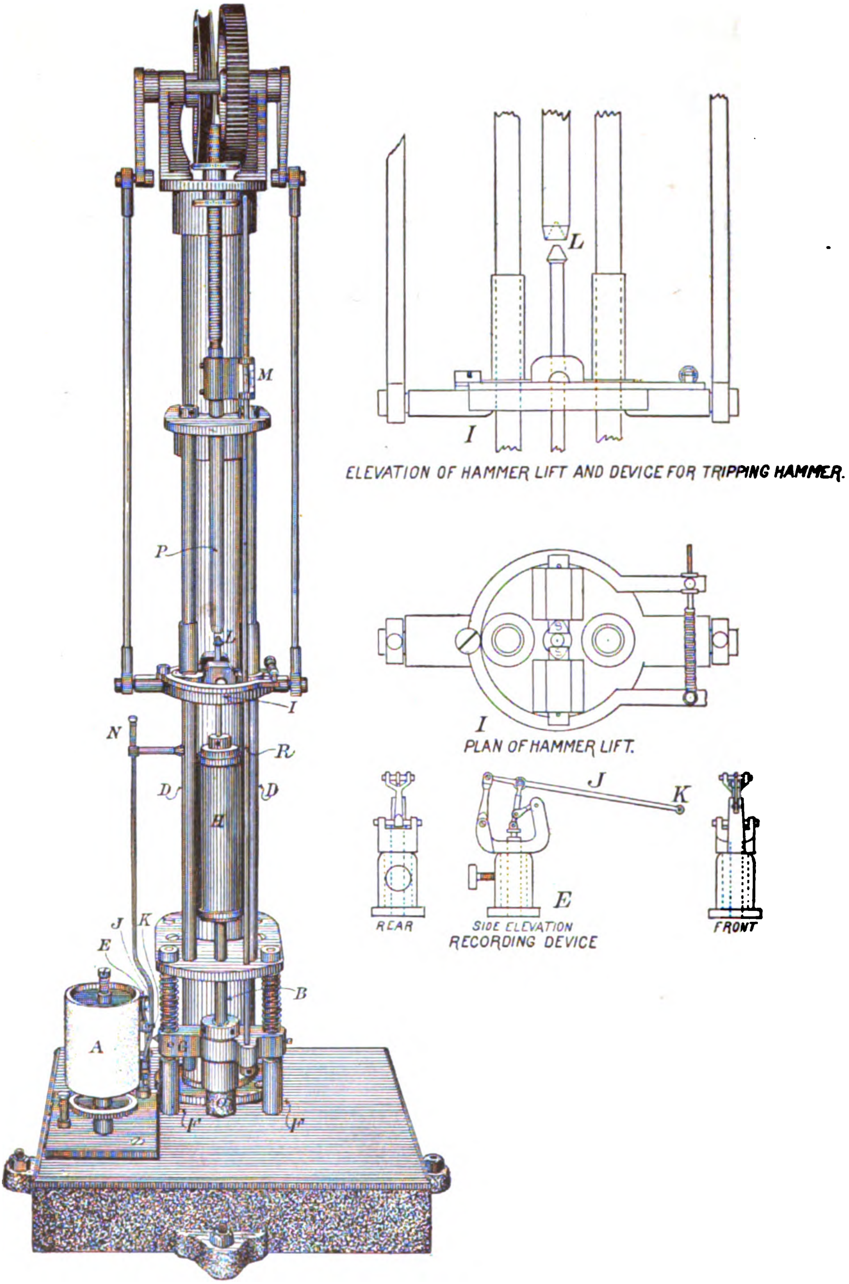


FIG. 5.—Briquette machine.

exactly centered and brought free of the guide rods (*D*). As the cross-head (*I*) continues to rise, the bolts supporting the hammer, which are tapered at an angle of about 45°, are thrust open by the sloping head of the adjusting cone rod (*P*) releasing the hammer, which falls on



ELEVATION OF HAMMER LIFT AND DEVICE FOR TRIPPING HAMMER.

PLAN OF HAMMER LIFT.

REAR

SIDE ELEVATION

RECORDING DEVICE

FRONT

FIG. 6.—Impact machine to determine cementing value of rock.

a flat-end plunger (*B*) of 1 kg weight, which is pressed upon the briquette (*O*) by two light spiral springs surrounding the guide rods (*F*). This plunger (*B*) is bolted to a crosshead (*G*). A small lever (*J*) holding a brass pencil (*K*) at its free end, is connected with the side of the crosshead, by a link motion arranged so that it gives a vertical movement to the pencil five times as great as the movement of the crosshead. The pencil is pressed against a drum (*A*), and its movement is recorded on a slip of silicated paper fastened thereon. The drum is moved automatically through a small angle at each stroke of the hammer; in this way a record is obtained of the movement of the crosshead during and after each blow of the hammer. To the crosshead (*G*) is fastened a steel rod (*R*) which passes up through the crosshead (*I*) and through a piece of metal securely attached to the cone rod (*P*). At this junction a vernier scale is graduated, by means of which the height of blow of the hammer can be accurately set to 0.1 mm, and by lowering the cone rod until it rests on the hammer cone (*L*) the height of the briquette can also be measured to 0.1 mm.

The standard fall of the hammer for a test is 1 cm (0.39 inch) and this blow is repeated until the bond of cementation of the material is destroyed. The blow producing failure is easily ascertained, for when the hammer falls on the plunger, if the material beneath it can withstand the blow it recovers; if not, the plunger stays at the point to which it is driven, and in either case the behavior of the test piece is recorded on the drum. The automatic record thus obtained from each briquette is filed for future reference. A copy of one of these records is shown in fig. 7. The number of blows required to destroy the bond of cementation or resilience, as described above, is noted and the average obtained upon five briquettes is given as the cementing value.

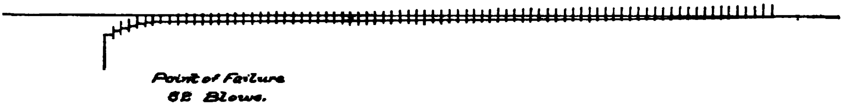


FIG. 7.—Diagram showing point of failure in cementation test.

The problem of holding the test piece rigidly under the intervening plunger, so that it would not be subjected to lateral movements and transverse strains, is one which has given much difficulty. Until recently a small brass plate with a beveled hole slightly larger than the diameter of the briquette was used, but it was found that the test piece was often seriously abraded by the side thrust developed. Later attempts to secure the briquette by various clamping devices were not satisfactory. Finally the method was adopted of placing a drop of thick shellac on the bottom of the test piece, which caused it to adhere firmly to the bedplate. Careful attention to such details as these is necessary in order to get satisfactory results from this test.

The original method for molding the briquettes was worked out in the laboratory of the Massachusetts Highway Commission and differs somewhat from the method as described above. In the earlier practice the requisite amount of rock dust to make a briquette was weighed out while dry, mixed with 3 to 4 cubic centimeters of water, and the briquette immediately molded from the wet dust. It is well known to practical road builders that the binding power of many rocks increases under the combined influence of water and traffic as time goes on. This question has received a great deal of attention and investigation in this laboratory. Experiments have shown that the cementing value is increased if the dough made from a rock dust is allowed to stand for some time before being molded, and it is still more increased if the dough is kneaded. This is plainly seen in the results obtained on a sample of dolomite. This dolomite is a celebrated road-building material in the locality in which it is found, and its most marked characteristic is the way that its binding power increases after it has been on the road for some time. A series of experiments was conducted, bearing on this point, in which the rock dust was made to a stiff dough and briquettes molded at stated intervals. A new lot of dust was made up and well kneaded by hand for one hour. The results were as follows:

	Cementing value.
Dough tested at once	16
4 hours old	50
24 hours old	81
72 hours old	79
96 hours old	77
120 hours old	79
144 hours old	83
8 days old	81
Kneaded 1 hour	190

An inspection of this table clearly shows the importance of conducting the operations of this test under uniform conditions. In mixing the dust with water to form the dough, the operator should be trained to knead each specimen as nearly as possible in the same manner for the same length of time. After twenty-four hours standing in a closed jar a cementing value is developed which remains fairly constant. When carried out in a uniform manner by a skilled operator this test has no higher percentage of variation on homogeneous samples than is found in testing the tensile strength of cement briquettes. Road materials vary widely in this important property, but the test is undoubtedly of the highest comparative value and practicability, for it distinguishes between excellent, good, fair, and poor binding materials. It should be remembered that from the very nature of the materials to be tested it is impossible to expect a degree of accuracy such as is obtained in the testing of perfectly homogeneous substances.

The maximum and minimum results obtained from the tests of the cementing value of the various materials received in this laboratory are given in the following table, and in Table A of the Appendix are found the results obtained on each sample.

TABLE III.—*Cementing values of road-building rocks.*

Name of rock.	Number of samples.	Maximum.	Minimum.	Name of rock.	Number of samples.	Maximum.	Minimum.
Amphibolite...	6	36	3	Gravel.........	31	725	2
Andesite.......	3	337	14	Gneiss.........	14	21	1
Basalt..........	9	72	2	Gabbro........	5	131	12
Chert	16	1,083	10	Granite........	25	77	2
Clay	13	4,000+	35	Limestone.....	52	231	8
Conglomerate..	3	327	12	Peridotite	1	3	3
Diabase........	26	110	2	Quartzite......	7	47	2
Diorite.........	9	137	1	Rhyolite.......	9	577	1
Dolomite.......	15	161	2	Sandstone.....	14	323	8
Eclogite	2	20	3	Schist	18	272	2
Felsite.........	7	101	2	Shale..........	3	985	62
				Slag	2	137	19
Field stone (erratics)	48	46	5	Slate	2	202	151
Flint...........	1	31	31	Syenite........	2	11	2

TEST FOR TOUGHNESS.[a]

This test is made on 25 mm × 25 mm (0.98 inch) rock cylinders with an impact machine especially designed for the purpose (fig. 8). Instead of a flat-end plunger resting on the test piece as in the cementation test, a plunger with the lower and bearing surface of spherical shape, having a radius of 1 cm (0.4 inch) is used. It can be seen that the blow as delivered through a spherical-end plunger approximates as nearly as practicable the blows of traffic. Besides this, it has the further advantage of not requiring great exactness in getting the two bearing surfaces of the test piece parallel, as the entire load is applied at one point on the upper surface. The test piece is adjusted so that the center of its upper surface is tangent to the spherical end of the plunger, and the plunger is pressed firmly upon the test piece by two spiral springs which surround the plunger guide rods. The test piece is held to the base of the machine by a device which prevents its rebounding when a blow is struck by the hammer. The hammer weighs 2 kg and is raised by a sprocket chain and released automatically by a concentric electro-magnet. The test consists of a 1 cm fall of the hammer for the first blow, and an increased fall of 1 cm for each succeeding blow until failure of the test piece occurs. The

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[a] This test was designed by the writer while in charge of the laboratory of the Massachusetts Highway Commission, but was never put in operation. It has been very much modified in the Washington laboratory.

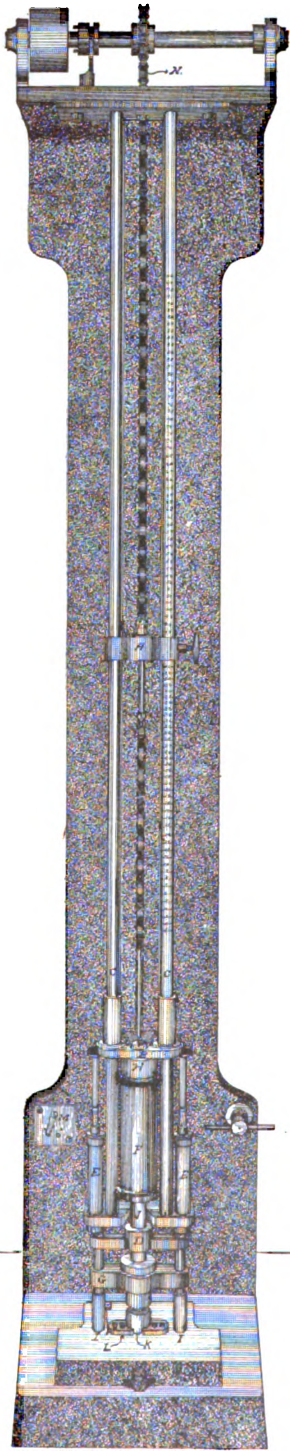

Fig. 8.—Impact machine for determining toughness.

number of blows required to destroy the test piece is used to represent the toughness. A sufficient number of results have not yet been obtained with this test to warrant their publication.

The cylindrical test pieces employed are made with a core saw designed for the purpose. It consists of a steel tube supported by journals in a vertical position and held in a cast-iron frame. A link motion lever rests on the upper end of the tube on a ball bearing. A spiral spring is attached to the free end of the lever by which the downward pressure of the tube can be adjusted. Water is fed in the top of the tube by a small rubber hose, and at the center is a pulley wheel by which the tube is revolved at about 800 revolutions per minute. The bottom or cutting end of the tube is set with bort by a specially designed method. This saw cuts very rapidly and the waste is very much less than in cube cutting, in addition to which the cylindrical test piece is better adapted for this test.

ABSORPTION TEST.

The method used for determining the absorptiveness of rock is not intended to give the porosity but merely the number of pounds of water that is absorbed by a cubic foot of rock in ninety-six hours. A smoothly worn stone, between 20 and 60 grams in weight, which has been through the abrasion test is used. After being weighed in air, it is immersed in water and immediately reweighed in water. After ninety-six hours of immersion it is again weighed in water. The absorptiveness of the rock is expressed by the following formula:

Number of pounds of water absorbed by a cubic foot of rock=

$$\frac{C-B}{A-B} \times 62.5$$

in which A is equal to the weight in air, B the weight in water immediately after immersion, C the weight in water after absorption for ninety-six hours, and 62.5 the weight of a cubic foot of water. From these weights the specific gravity and the weight per cubic foot of the rock are also determined.

The method first used by Gilmore,[a] and later by Winchell and Williams,[b] differs from this method in that the stone used in the test was taken out of the water in which it was immersed, its surface dried with a filter paper, and its weight taken every twenty-four hours until absorption ceased. The absorptiveness was expressed by giving the number of parts by weight of stone required to absorb one part by weight of water. The method used in this laboratory is in no way better than the earlier method except that it requires less manipulation and the results are more practical for road builders. Table I, on page 13, shows the maximum and minimum per cent of water absorbed by each variety of rock tested and has already been considered in connection with the effect of freezing on road materials.

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[a] A. A. Gilmore. Report on Building Stones of the United States, 1875, p. 8.

[b] J. F. Williams. Annual Report of the Arkansas Geological Survey, 1890, p. 47.

SPECIFIC GRAVITY AND WEIGHT PER CUBIC FOOT.

The specific gravity and weight per cubic foot for all rock samples are calculated from the weights used for determining the absorptiveness of rocks, which are given in Table A of the Appendix.

TEST FOR HARDNESS.

The laboratory is equipped with a Dorry machine for testing the hardness of materials according to the standard method of the French School of Roads and Bridges. The machine, however, has just been installed and no results have as yet been obtained.

COMPRESSION AND TENSILE STRENGTH TESTS.

The laboratory is equipped with a 200,000-pound universal Riehlé testing machine and a 30,000-pound Olsen. Although compression and tensile strength tests do not form part of the routine examination of road materials, they are often of the highest value in special cases.

TESTS FOR PAVING BRICK.

RATTLER TEST.

The machine for conducting this test is shown in fig. 9. It consists essentially of a rotation drum partially filled with large and small cast-iron blocks or shot. The wearing quality of paving brick is judged by the loss sustained after a sample has been subjected in this drum for one hour to the impact and abrasion of these cast-iron shot. For the first 600 rotations the loss is mainly due to a chipping action which develops the brittleness of the brick; between 1,200 and 1,800 rotations the loss is mainly due to an abrasive action which develops the softness of the brick. During the test any defects of structure, such as planes of separation, will probably result in rupture. The particular form of rattler shown was designed and built at Purdue University, Lafayette, Ind. It is an overhanging rattler, and at the free end there is convenient access to the drum for the purpose of inserting and removing the bricks, an operation which in the ordinary form of rattler necessitates the removal of a slat of the drum. A tight sheet-iron cover prevents the escape of dust. The worn material is caught in a pan, as shown in the illustration.

The rattler is 28 inches in diameter and 20 inches long and has 14 cast-steel slats. It is run at a speed of 30 rotations per minute for 1,800 revolutions. The charge to be placed in the rattler consists of 9 paving blocks or 12 dry bricks, together with 300 pounds of shot made of ordinary machine cast-iron. The shot are of two sizes. The larger size (2½ by 4½ inches) weigh 7.5 pounds each and have slightly rounded edges; the smaller shot (1.5-inch cubes) weigh about seven-eighths of a pound each. The charge is composed of 75 pounds of the larger and 225 pounds of the smaller size. The individual shot are replaced by new ones when they have lost about one-tenth of their original weight. The loss in weight undergone by a sample of the brick under test at the end of 1,800 revolutions is found by comparing the original weight of the brick with its weight after test. The loss in per cent of the original weight is the factor determining the quality of the brick. For the best paving brick this loss should not be in excess of 18 per cent. The above conditions are those demanded in the standard specifications recommended by the National Brick Manufacturer's Association.

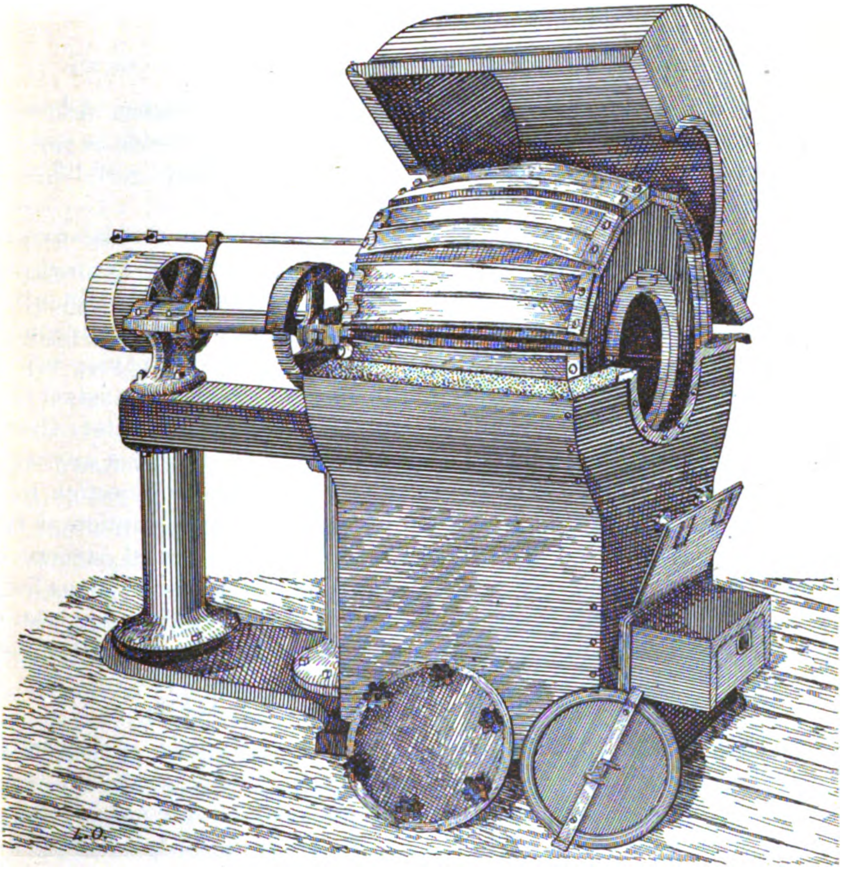

FIG. 9.—Brick rattler.

In order to collect data which will give a knowledge of the degree of variation in the quality of individual brick this laboratory determines the loss of weight of individual brick as well as the average loss of the charge. Furthermore, since the loss during the earlier stages of the test is mainly due to a chipping action and the subsequent loss to the wearing or abrasive action of the charge, both factors of quality are reported, viz, the chipping loss at 400 or 600 rotations and the wearing loss between 1,200 and 1,800 rotations. The form of report for the rattler test is given in the Appendix.

The rattler test for paving brick has won the confidence of engineers after a very searching investigation extending over a number of years. As now conducted, this test is constant in its action and gives valuable evidence concerning the wearing qualities of brick.

ABSORPTION TEST.

The absorption test is valuable in determining the degree of heat at which the brick was burnt. It is not, however, practicable to specify a fixed per cent of absorption to which competitive samples of different brands of paving brick must conform.[a]

The absorptiveness of paving brick is determined in this laboratory by immersing in distilled water a brick which has passed through the rattler test and been thoroughly dried. The gain in weight after ninety-six hours' immersion is determined and expressed in percentage of the original weight of the brick. The fact that some paving bricks are covered with an impervious shell makes it desirable to conduct this test on a brick whose interior structure is accessible to water; therefore, only bricks that have been subjected to the rattler test are used. As far as present evidence shows, there is no serious objection to a high rate of absorption on account of danger from disruption of the brick through the action of frost. Defects of internal structure, however, such as internal planes of division due to faulty annealing, present an opportunity for the disruptive action of freezing water. These internal faults of structure are made evident in the rattler test.

CROSS-BREAKING AND COMPRESSION TESTS.

Facilities exist in the laboratory for performing both of these tests according to approved methods on the testing machines already mentioned. The core drill shown in fig. 10 is available for making cylindrical test pieces of 1 or 2 inch diameter from the body of the brick for the compression test. Cross-breaking and compression tests are, however, considered to be of secondary importance and do not furnish as valuable evidence of the quality of the brick as do the rattler and absorption tests.

TESTS FOR CEMENT.

For making physical and mechanical tests on cement a Fairbanks testing machine and other necessary equipment are used. The standard methods adopted by the American Society of Civil Engineers are closely followed. Facilities also exist for the analysis of the raw materials and their actual mixture and manufacture into cement on a laboratory scale. Lack of funds, however, has so far prevented the testing or investigation of cements and concretes except in a desultory way.

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[a] Seventeenth Annual Report of the Illinois Society of Engineers and Surveyors, 1902, p. 160.

CLASSIFICATION OF MATERIALS.

It is most essential that all rocks tested at the laboratory should be analyzed petrographically or chemically, and properly classified, otherwise endless confusion would result.

PETROGRAPHIC ANALYSIS.

The methods of petrographic examination do not differ essentially from those generally employed. The laboratory is equipped with an exceptionally good microscope of the latest Fuess model, which, besides the usual attachments, is provided with a revolving analyzer in the tube for the determination of very low doubly refracting minerals and a Schwarzmann scale for the measurement of optical axial angles. Another important accessory is the detachable-screw micrometer, movable in the focal plane of the ocular by means of a drum screw, which, with the most powerful objective lens, records a drum interval of 0.000040 mm.

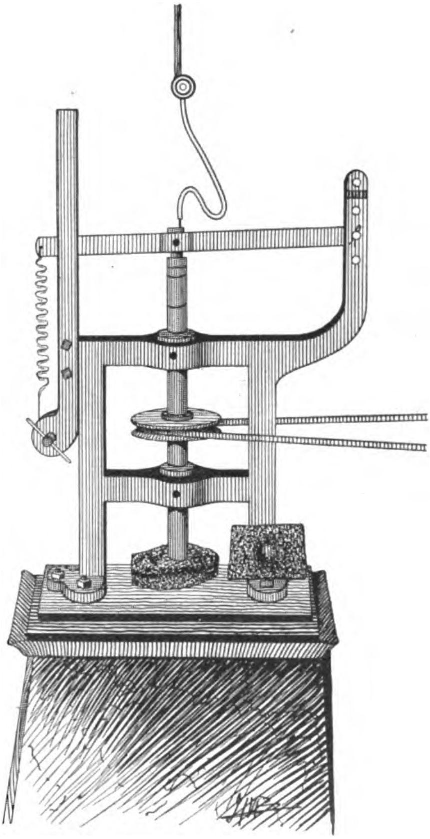
FIG. 10.—Core drill.

A very important feature of the microscopic work is the relative quantitative determination of the mineral components of rock, to accomplish which an ordinary fixed eyepiece having a square field was divided into 100 quadratic areas. With the aid of this cross-line field, each square of which is one one-hundredth of the whole field, the relative proportions of the minerals occupying the field can be readily determined by simply noting the number of squares covered by each mineral in turn. Averages derived from numerous examinations of this kind in various parts of the section indicate the percentage of the different minerals constituting the rock itself.

In describing the character of the material, the origin of the rock, as far as revealed by the microscope, has been observed. Thus rocks with well-defined porphyritic or flow structure, such as rhyolite, andesite, volcanic tuffs, etc., are considered as effusive or volcanic, while deep-seated varieties such as granite, syenite, quartz-porphyry, etc., are described as plutonic or intrusive as the case may be. When the mode of origin is obscure, the general term "igneous" is used. Sandstones, limestones, etc., on the one hand, and quartzites, schists, gneisses, etc., on the other are treated as sedimentary and metamorphic rocks respectively, while disintegrated material such as sands, clays, etc., are classed as sedimentary deposits.

In describing the igneous rocks a very simple classification according to well-recognized types has been best adapted to the requirements of the laboratory. No attempt is made to subdivide these main families except in rare instances where minerals foreign to the normal composition of the rock occur in appreciable quantities. Thus a granite rich in hornblende would be termed hornblende-granite, or a syenite containing much augite, augite-syenite, etc.

Finally the reports under general remarks call attention to the origin of the secondary constituents as well as to the color, texture structure, and other physical properties of the rock specimen likely to be of use to road builders. The tabulated results on all samples examined will be found in Table B of the Appendix. Microphotographs have been made from sections of some of the principal types of rocks examined and some of these photographs, selected to show the characteristic structure of various types, are reproduced in Plates I to V, inclusive.

CHEMICAL ANALYSIS.

The purpose of the chemical analysis of road materials is to supplement the petrographic identification and classification of rocks as well as to supply data which when taken in connection with the physical tests may furnish information as to the value of a given material. The study and chemical examination of clays, marls, etc., has become an important part of the laboratory work, not only because the manufacture of cements, drain tiles, and pipes is indirectly a road-building problem, but also on account of recent efforts to construct practical roads by mixtures of burnt and raw clay in sections of the country where no stone is available. The rapid growth of intelligent interest, which is taking place all over the country, in the problems of road building has brought about a greatly increased demand for laboratory methods of investigation. The number of samples presented for examination and the subsidiary value of the chemical examination in most cases have led to modifications of the ordinary methods of rock analysis which, in the experience of this laboratory, furnish a sufficient degree of accuracy with the minimum expenditure of time.

The number of inquiries received as to the methods used in the chemical analysis of rocks, clays, cements, etc., has shown that in spite of the great number of text books on technical chemical analysis, there is a demand for methods of rock and clay analysis which shall be at the same time practical, rapid, and accurate enough for the purpose in hand.

The methods as given in detail in the Appendix have been tested in the hands of several workers on a large number of samples, and have been found to yield satisfactory results when compared with more complete and accurate work. Analysis is always made on air-dry samples, as the directions usually given in text-books for operating on samples dried at 100° C. invariably lead to bad results, since plastic clays and rock powders reabsorb comparatively large quantities of water from the air after being heated.[a]

By following the scheme of analysis given in the Appendix several analyses can be completed by one man in one working day, provided the fusions have been made on the preceding day and the evaporation for the separation of the silica allowed to go on over night. There is no claim made that these short cuts yield results as accurate as more precise and tedious methods; in fact, any scheme of analysis which divides a gram sample into five aliquot parts is open to criticism, as each determination, with the exception of the silica, has its error multiplied by five. Nevertheless, if the pipette for dividing the solution is accurately standardized to deliver exactly the fifth of the contents of the half-liter flask used, the summation of the complete analysis comes within 0.5 per cent of 100 in the large majority of analyses.

Among the rarer or more unusual elements only manganese and titanium are included in the scheme of analysis. Undoubtedly other elements, such as chromium, barium, strontium, zirconium, lithium, chlorin, and fluorin are present in many rocks and clays, but usually the amounts are so small that for practical purposes they can be ignored. Titanium, on the other hand, is rarely absent and is not infrequently present to the extent of several per cent. Although the effect, if any, of titanium on the properties of clays and cements is not known, it is certainly wrong to report this constituent as alumina, as is nearly always done.

THE APPLICATION OF LABORATORY RESULTS TO PRACTICE.

The proper interpretation and application of the results obtained in the laboratory are quite as important as the general accuracy and appropriateness of the tests themselves. It is probable that many engineers and others interested in the subject of road building who have found time to examine the question only superficially have misunderstood the bearing and value of road-material testing. In all cases the results obtained in testing materials of construction are of relative rather than absolute value. Even quite a large variation in the results yielded by different test pieces of the same sample should not condemn the practical value of the figures if they are properly applied and interpreted. The necessary qualities of road materials have to be considered from the double standpoint of furnishing a strong, unyielding, well-drained road foundation and a hard, coherent binder surface. In road building the attempt should be made to get a perfectly smooth surface, neither too hard, too slippery, nor too noisy, and as free as possible from mud and dust, these results to be attained and maintained as cheaply as possible. Such results, however, can be achieved only by selecting the material and methods of construction best suited to the conditions.

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[a] Cushman. Jour. Am. Chem. Soc., May, 1903.

Given a number of materials for laboratory examination, it is not pretended that an actual practical grade of excellence can be established. On the other hand, if more than one material is available, it is quite possible for the laboratory to point out which one would yield the best results both as to immediate excellence and length of life under known conditions of climate and traffic. Undoubtedly in many cases large sums of money have been wasted in building roads from unsuitable material, which might have been saved by referring the materials to the laboratory. If, for instance, it is desired to know whether an available rock will be useful as a top dressing to form the binder surface, no better method of obtaining preliminary information on the subject is known than to test the cementing value. Undoubtedly some rocks will yield powder from successive test pieces which shows very wide variations in this value under the conditions of the test, but there is at present no difficulty in distinguishing between good and bad material. It is true that before the discovery that certain road materials increase in binding power after being in use for a time some errors were made. A notable, although exceptional, example of this kind was a southern chert from Alabama. The method formerly used of mixing the rock dust with water and immediately molding the briquettes, as previously described in this bulletin, yielded a low cementing value, and the rock was not recommended as a surfacing material if a better one was available. It appears, however, that these southern cherts develop a high binding power under traffic and make excellent roads. Under the present method of conducting the cementation tests—i. e., allowing the dough to stand before molding it, the briquettes of this same chert gave a comparatively high cementing value. It has been impossible, under the pressure of routine work during the current year, to prepare the figures and results of these and many other investigations for publication in this bulletin. These results will be reserved for a special report on the cementing value of road materials, to be published as soon as possible.

It has already been stated that a road material can not be selected, irrespective of the volume and character of traffic and the climatic conditions to which it is to be subjected, without the risk of failure. When the very great cost even of rural highways is considered, it is evident that it is gross negligence not to use every preliminary precaution to guard against expensive mistakes. In order to properly designate the conditions best suited for a particular material it soon became evident that traffic must be classified according to its volume and character. The following five divisions of traffic are therefore made: City (perurban), urban, suburban, highway, and country road. City (perurban) traffic is such as exists on the business streets of large cities, and no macadam road can withstand it. For such a traffic stone and wood blocks, asphalt, brick, or some such materials are necessary. Urban traffic is such as exists on city streets which are not subjected to continuous heavy teaming, but which have to withstand very heavy wear, and require the hardest and toughest macadam rock, or other highly resistant material. Suburban traffic is such as is common in the suburbs of a city and the main streets of country towns. Highway traffic is a traffic equal to that of the main country roads. Country road traffic is that of the less frequented country roads.

This classification is purely arbitrary, but it serves the purpose for which it was intended, and each group can be approximately identified by a road builder. City (perurban) traffic requires the hardest and toughest materials available, having the highest wearing qualities. For urban traffic, such materials as asphalt, brick, wood block, and bituminous macadam are suitable, and if ordinary macadam is used, a rock of the highest hardness, toughness, and wearing qualities is needed. For a suburban traffic the best rock would be one of high toughness but of less hardness than for urban traffic. For highway traffic a rock of medium hardness and toughness is best, and for country road traffic a comparatively soft rock of medium toughness.

To obtain the best results on a macadam road that rock should be selected which would resist the wear of the traffic to which it is subjected to such a degree as to supply just the sufficient amount of binding material to cement the road. Too much or too little wear are alike injurious. The higher the cementing value of the rock the smaller the wear necessary. When a road is first constructed a sufficient amount of binding material must be supplied to cause it to "come down" under the roller or traffic. If the subsequent traffic is not sufficient to wear off the requisite amount of binder to replace that carried off by wind and rain the road "ravels." If the traffic wears off an excess of binding material, mud and dust result. In either case the material is not well suited to the conditions. In the first case a softer rock should be used; in the latter, a harder, tougher rock, and in all cases a rock of high binding power. For example, if a country road or city park way, where only a light traffic prevails, were built of a very hard and tough rock with a high cementing value, neither the best results nor the cheapest, if a softer rock were available, would be obtained. Such a rock would so effectively resist the wear of a light traffic that the amount of fine dust worn off would be carried away by wind and rain faster than it would be supplied by wear. Consequently the amount of binder produced by wear would be insufficient, and if not supplied from some other source the road would soon go to pieces. The first cost of such a rock would in most instances be greater than that of a softer one, and the necessary repairs resulting from its use would also be expensive.

It has already been pointed out in a former publication[a] from this laboratory that rocks belonging to the same species and having the same name, but coming from different localities, vary from each other in their physical road-building properties almost as much as they do from rocks of distinct species. Wide variation also occurs in the mineral composition of rocks of the same species, as well as in the size and arrangement of their crystals. It is impossible, therefore, to classify rocks for road building by simply giving their specific names. It can be said, however, that certain species of rocks possess in common some road-building properties. For instance, the trap[b] rocks as a class stand heavy traffic well, as they are hard, tough, usually have binding power, and are therefore frequently spoken of as the best rocks for road building. This, however, is not always true, for numerous examples can be shown in which trap rocks having the above properties in the highest degree have failed to give good results on light-traffic roads. The reason trap rock has gained so much favor with road builders is because a large majority of macadam roads in our country are built to stand an urban traffic, and the traps stand such a traffic better than any other single class of rocks. There are, however, other rocks that will stand an urban traffic perfectly well, and there are traps that are not sufficiently hard and tough for a suburban or highway traffic. The granites are generally brittle and many of them do not bind well but there are a great many which when used under proper conditions make excellent roads. The felsites are usually very hard and brittle and many have excellent binding power, some varieties being suitable for the heaviest macadam traffic. Limestones vary greatly but generally bind well; they are soft and frequently improve under traffic. Quartzites are almost always very hard, brittle, and have very low binding power. The slates are usually soft, brittle, and lack binding power.

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[a] The Selection of Materials for Macadam Roads, Yearbook of the Department of Agriculture, 1900.

[b] This term is derived from the Swedish word *trappa*, meaning steps, and was originally applied to the crystallized basalts of the coast of Sweden, which much resemble steps in appearance. As now used by road builders, it embraces a large variety of igneous rocks, chiefly those of fine crystalline structure and of dark-blue, gray, and green colors. They are generally diabases, diorites, trachytes, and basalts.

When samples of rock are received at the laboratory without a request for specific information, the method of procedure is as follows: The samples are subjected to the tests already enumerated, and the results are classified and compared with those of like value and known worth in practice. These results, together with a statement of the particular group of traffic to which the materials are best suited are forwarded to the sender[a]. When samples are sent for the purpose of ascertaining which is best suited for a particular road, the sender is requested if possible to supply the laboratory with a census of the traffic or, if this is impracticable, to state what class of traffic passes over the road. The tests are then made and the results classified and compared as in the previous case. Full information regarding the climatic conditions is obtained from the Weather Bureau and that material is selected which the results indicate to be best suited to the conditions. Allowance is always made for at least a 15 per cent increase in the volume of traffic due to the improved conditions, if the road is to be macadamized for the first time.

As time goes on and the behavior of these tested materials is studied and records of traffic are accumulated the value of the laboratory tests will constantly increase. Cooperation among the increasing number of laboratories in the country is much needed and uniform methods of testing should be adopted. The American Society for Testing Materials has been asked to appoint a committee to investigate the whole subject of the testing of road materials with a view to bringing the work of the various and widely separated laboratories under standard conditions. As soon as the rapidly growing mass of data on this subject is thus made uniform and comparable it will be of the very highest value.

Those who are familiar with the problems of rural road building know the great difficulty of selecting among the available materials for a particular road the one which will give the best results for the least cost of construction and maintenance. There are, undoubtedly, practical road builders whose judgment on the road-making quality of a rock is excellent, but experience with materials of construction in general has proved that it is wise and economical to test the physical properties of materials before entering on the expenditure of large sums of money. Bridge building would not have become the high art that it has had not the careful and systematic testing of materials put into the hands of the engineer preliminary data on which to base his calculations and estimates. It is hoped that before long intelligent cooperation will make this true of road building. To this end the laboratory has decided on the following plan to govern its future work. It will require a few years for the completion of the plan, but by following it until a better one is evolved the usefulness of the laboratory should constantly increase.

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[a] See forms on page 75 of the Appendix.

As it is impossible to accurately judge the quality of a road material unless the conditions of climate and traffic on the particular road where it is to be used are known, it is intended to secure such data as far as possible. For this purpose a form[a] for making a census of the volume and character of traffic on any road has been prepared. The cost of such a census is trifling when the total cost of the road is considered, and the observations may be made by anyone living within view of the road. When enough of these records have been accumulated, the traffic of a road may be expressed by a number, if a unit and standard scale are agreed upon to suit the average conditions of the country as has been done in France. The maximum, minimum, and mean conditions of temperature, rainfall, and wind velocity for any locality can be obtained from the Weather Bureau, and also classified according to a similar scale. When a large number of standard results of different tests have been accumulated and compared with the results of practice, it will be possible to classify them into five groups to correspond to the five subdivisions of traffic. Thus, when a test sample is received at the laboratory, together with a census of the traffic on the road to be constructed, the traffic and climate will be placed in their proper groups and the hardness, toughness, resistance to wear, cementing value, weight per cubic foot, and absorptiveness will each be graded according to the same standard scale. By this method the best available material for any road can be selected with a high degree of accuracy, for the proposed classification will be based on the observations of practice as well as on laboratory tests. Such results can not fail to be of the highest value to the road engineer.

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[a] See page 77, Appendix.

APPENDIX.

METHODS OF CHEMICAL ANALYSIS ESPECIALLY ADAPTED TO THE RAPID EXAMINATION AND CLASSIFICATION OF ROAD MATERIALS.

I. COMPLETE ANALYSIS.

A.—*Substances mainly insoluble in hydrochloric acid.*

The sample for analysis is powdered on the agate mortar until all indication of gritty particles has disappeared. The sample is then spread in a thin layer between filter papers and allowed to stand overnight to air dry.

Exactly 1 gram is weighed into a large platinum crucible and mixed with from 4 to 6 grams of a fusing mixture, which is made by grinding together in a porcelain mortar 50 grams sodic carbonate, 50 grams potassic carbonate, and 2 grams of potassic nitrate.

The crucible is heated up gradually over an ordinary Bunsen flame until all bubbling has ceased and the contents are in a state of quiet fusion, when the blast lamp is applied at its maximum temperature for ten minutes. The liquid fusion is immediately poured into a platinum dish floating in a basin of water. The material still adhering to the crucible is dissolved in dilute hydrochloric acid with the usual precautions, and after the removal of the crucible and cover the fused button is added. After solution is complete the cover is removed from the casserole and the contents evaporated to dryness as quickly as possible on the steam bath. As dryness is approached the lumps are broken up with a button-ended stirring rod, and the heating continued until the residue has the consistency of a dry powder. This residue is taken up with dilute hydrochloric acid and the silica filtered and washed. The procedure at this point depends upon the degree of accuracy desired, and two methods are accordingly suggested.

(1) *Accurate method.*—The filtrate from the silica is again evaporated to complete dryness, taken up with dilute hydrochloric acid, and the small amount of silica filtered out, washed, and united with that obtained by the first filtration. After burning off the paper the white silica is blasted at the highest temperature of the blast lamp for fifteen minutes, cooled, and weighed. It is then treated with an excess of hydrofluoric acid and two or three drops of strong sulphuric acid, evaporated carefully to dryness, and finally blasted. The true per cent of silica present is shown by the loss in weight. The weighed crucible containing the small residue is preserved for the ignition later on of the main portion of the alumina, together with the oxids of iron, phosphorus, and titanium, but as the filtrate is divided into aliquot parts, only one-fifth of the weight of the residue applies as a correction.

(2) *Rapid approximate method.*—One evaporation to complete dryness on the water bath is made, care being taken to break up the lumps of the residue by stirring and rubbing with a flat-end glass rod as the point of dryness is approached. The residue, which should be quite dry and powdery, is taken up with hydrochloric acid and the

silica filtered, washed, and burned white over an ordinary Bunsen flame. The error resulting from a single evaporation on the water bath is in most cases very nearly counterbalanced by the error introduced by omitting the blasting, while the saving in time is very considerable. The slight amount of silica which remains in solution is carried down later with the iron and alumina, and serves to counterbalance the small quantity of aluminum phosphate and alumina almost always found in the silica.

Whichever of the two procedures as given above has been followed, the filtrate from the silica is transferred to a 500 cc graduated flask, diluted to the mark and very thoroughly shaken. This is solution A. A 100 cc pipette which has been carefully calibrated to divide the contents of the flask into exact fifths under standard conditions is used for dividing the solution into aliquot parts. Manganese usually shows itself in the green color which it imparts to the fusion, and a slight pinkish color which can be observed when the fusion is first treated with hydrochloric acid. A 100 cc portion having been transferred to a beaker, its treatment will depend upon whether or not manganese is present.

(1) *When manganese is present.*—Add a solution of sodic carbonate until one or two drops in excess have produced a permanent precipitate. Clear with one drop of concentrated hydrochloric acid. Dilute to 200 cc, add ten drops of glacial acetic acid and 2 grams of crystallized sodic acetate. Heat to boiling and boil three minutes. Allow to settle and filter hot. Wash a little with 2 per cent sodium acetate solution, dissolve the precipitate in an excess of hydrochloric acid, and reprecipitate with ammonia—filter, wash, ignite, and weigh. The two filtrates are combined and boiled for some time with additions of bromin water until the precipitation of manganese dioxid is complete. The precipitate is ignited over the ordinary Bunsen flame and weighed as Mn_3O_4. The filtrate from the manganese dioxid precipitate which contains calcium and magnesium is put on the steam bath for concentration while other portions of the analysis are proceeded with.

(2) *When manganese is absent.*—Ammonia is added in slight excess, the solution boiled for two minutes and filtered hot, using the pump. Unless the precipitate is a small one, another precipitation is necessary. The precipitate is dissolved in hydrochloric acid in the same beaker, reprecipitated with ammonia, filtered, washed with hot water, blasted, and weighed. If necessary the combined filtrates are placed on the steam bath for concentration. The paper containing the precipitate is transferred wet to a weighed crucible. If the accurate method has been used, there will be a weighed crucible ready to receive it. The paper and contents are rapidly dried by keeping the crucible moving over a free flame, the paper is burned off and the precipitate finally blasted and weighed. This weight represents ferric oxid, alumina, titanium oxid, and phosphoric anhydrid.

TOTAL IRON.

One hundred cubic centimeters of solution A is poured directly through a reductor and followed by 150 cc of distilled water. The clear, colorless solution is transferred to a beaker, and to it is added 50 cc of dilute sulphuric acid and finally 5 cc of a 20 per cent solution of manganous sulphate. The contents of the beaker are immediately titrated with standard permanganate solution. The titration with bichromate is not nearly so rapid as the titration with permanganate and with very small amounts of iron the end point can not be accurately distinguished. A large number of trials has shown that this direct method of titration in the presence of hydrochloric acid is quite as accurate as those which call for an evaporation with sulphuric acid or a preliminary precipitation with ammonia, filtration, and re-solution in sulphuric acid. A permanganate solution is made by dissolving 1.975 grams in a liter. One cubic centimeter of this solution equals 0.005 gram ferric oxid (Fe_2O_3). The reductor used is practically the same as the simple device described by Shimer.[a] The tube has an internal diameter of three-eighths of an inch, and is 20 inches long. It is drawn out and cut off at its lower extremity with an opening of 1 mm diameter. One inch of coarse sand is placed at the bottom and the rest of the tube is filled with amalgamated zinc. The ordinary B and A "iron free" granulated zinc is moistened with dilute sulphuric acid and a little clear mercury stirred in. Suction is applied and regulated so that 60 cc of solution passes through the reductor in five minutes. The washing is allowed to proceed more rapidly. Blanks are run to determine the constant correction for the reductor.

TITANIUM.

To 100 cc of solution A, in a porcelain evaporating dish, 10 cc of strong sulphuric acid is added and the solution evaporated on the steam bath until the hydrochloric acid is expelled and the sulphuric acid solution has little or no yellow color. After slight dilution the solution is filtered into a 100 cc graduated cylinder, 2 cc of pure 3 per cent hydrogen peroxid[b] added, and the solution diluted to 50 cc. In another graduated cylinder containing about 40 cc of water, 2 cc of 3 per cent peroxid is added and then gradually a standard solution of titanium run in from a burette until the colors match, when the solutions are looked at through the same depth of column. The standard solution used in this laboratory is made up so that 1 cc equals 0.0002 gram of titanium oxid (TiO_2). The standard is made up by roughly weighing out somewhat more than the right amount of some titanium salt or compound, and after getting it into solution standardizing it by gravimetric analysis, subsequent dilution, and reanalysis.

PHOSPHORIC ACID.

The volumetric method based upon that adopted by the Association of Official Agricultural Chemists is used for the determination of phosphoric acid. The following solutions are prepared:

PREPARATION OF REAGENTS.

(1) *Molybdic solution.*—One hundred grams of molybdic acid are dissolved in 417 cc of ammonia, specific gravity 0.96, and the solution thus obtained poured into 1,250 cc of nitric acid, specific gravity 1.20. The flask in which the mixture is made is kept cool by immersion in water until the reaction is completed. The mixture is kept in a warm place for several days, until a portion heated to 40° C. deposits no yellow precipitate, when the solution is decanted and preserved in a glass-stoppered bottle.

(2) *Standard potassium hydroxid solution.*—This solution is made up to contain 3.6342 grams of potassium hydroxid to the liter. It is prepared by diluting 64.76 cc of normal potassium hydroxid, which has been freed from carbonates to 1 liter. One cubic centimeter is equal to 0.2 mg of phosphoric acid (P_2O_5).

(3) *Standard nitric acid solution.*—The strength of this solution is the same as the standard alkali solution, and is determined by titrating against that solution, using phenolphthalein as indicator.

(4) *Phenolphthalein solution.*—One gram of phenolphthalein is dissolved in 100 cc of alcohol.

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[a] Jour. Amer. Chem. Soc., **21**: 723.

[b] Mere traces of hydrofluoric acid, either in the peroxid or the titanium solution, render this method inexact, hence care should be exercised as to the character of the peroxid, which, as sold in the market, often contains fluorin. (Hillebrand, Bul. U. S. Geol. Sur. 176, p. 68.)

DETERMINATION.

One hundred cubic centimeters of solution A is put into an Erlenmeyer flask, made slightly alkaline with ammonia and then again acidified with nitric acid. About 10 grams of dry ammonium nitrate is added and the solution heated to 60° C. Twenty-five cubic centimeters of the molybdate solution is then poured into the flask, a smooth, tightly fitting rubber stopper inserted, and the contents of the flask vigorously shaken for five minutes. After allowing a short time for the yellow precipitate to settle it is filtered and washed five or six times with a 3 per cent solution of potassium nitrate. The precipitate and filter is transferred to a beaker, dissolved in a small excess of standard alkali, a few drops of phenolphthalein added, and the solution titrated with the standard nitric acid.

LIME.

The concentrated filtrate from the iron, alumina, etc., is made decidedly ammoniacal and brought to a boil, a sufficient quantity of saturated solution of ammonium oxalate is then added, avoiding a large excess. Boiling is continued for a few minutes when the beaker is immediately set into a dish of cold water. The precipitate settles rapidly, and is at once filtered, using a platinum cone with the pump, and washed thoroughly with hot water. The filtrate is reserved for the determination of the magnesia. The filter paper containing the washed calcic oxalate is lifted from the cone and opened inside a beaker. By means of a well-directed stream of a wash bottle all precipitate can be washed off the paper. Fifty cubic centimeters of dilute sulphuric acid is then allowed to run over the paper into the beaker, more water is added, the solution heated to about 60° C., and immediately titrated with standard permanganate solution. The lime factor is exactly one-half the iron factor of the permanganate solution.

MAGNESIA.

The filtrate from the lime determination is made slightly acid with hydrochloric acid and 30 cc of a concentrated solution of disodic phosphate added. It is then evaporated down to a bulk not exceeding 150 cc, transferred to an Erlenmeyer flask, and after cooling ammonia is added gradually with shaking until the reaction is strongly alkaline. The flask is stoppered with a smooth rubber stopper and shaken vigorously for five minutes. By this treatment the precipitate will usually be ready to filter inside of an hour. The filtrate should always, however, be preserved over night to make sure that no more precipitate appears. The precipitate is collected either on paper or a weighed Gooch crucible, burned, blasted, and weighed as magnesium pyro-phosphate ($Mg_2P_2O_7$), which multiplied by 0.3619 equals magnesium oxid (MgO).

SULPHURIC ACID.

Sulphur occurs in rock and clays usually as sulphids, more rarely as sulphates. The quantities are nearly always small, and in the cases where they are not the microscopic examination has disclosed the fact. It is customary in rock and clay analysis to report sulphur as SO_3, and that method has been followed.

The remaining 100 cc of solution A is heated to boiling, and a few cubic centimeters of a solution of barium chlorid added, and the boiling continued for five minutes. When the precipitate is settled it is collected on a paper or on a weighed Gooch crucible, washed with hot water, ignited, and weighed as barium sulphate ($BaSO_4$), which multiplied by 0.3430 equals SO_3.

FERROUS IRON.

The determination of iron in the ferrous state is an important matter in connection with the analysis of clays. Fairly satisfactory determinations can be secured by boiling 1 gram of the sample with hydrofluoric and sulphuric acids in a large covered platinum crucible. After boiling five minutes the solution is cautiously diluted and at once titrated with potassium permanganate.

POTASSIUM AND SODIUM OXID. (K_2O, Na_2O.)

One-half gram of the very finely ground powder is mixed with its own weight of pure ammonium chlorid, and the two thoroughly ground together. About 4 grams of pure calcic carbonate is added, and the grinding continued until an intimate mixture is made. This mixture is transferred to a J. Lawrence Smith tubular crucible [a] and the mortar rinsed with a little extra calcic carbonate. The crucible is capped and placed in a fire-clay furnace and heated for about ten minutes by a low flame. As soon as no more odor of ammonia is perceptible it is heated by the full heat of two Bunsen burners for three-quarters of an hour. After cooling, the sintered mass usually detaches from the crucible but it may be loosened with hot water. The mass is transferred to a platinum dish, and digested with hot water until completely disintegrated. The solution is filtered into a beaker and the residue washed, a few drops of ammonia and ten of a saturated solution of ammonia carbonate added, the solution filtered into a platinum dish, and the residue washed thoroughly. The filtrate is evaporated to dryness, and the residue heated at a dull red heat until the ammonia salts are expelled. After cooling, the residue is taken up with a little water, and a few drops of ammonia and ammonic carbonate added to make sure that all the calcium has been precipitated. The solution is filtered on a small filter into a weighed platinum dish, and the precipitate and paper washed with hot water. A few drops of dilute hydrochloric acid are added and the clear solution evaporated to dryness. The ammonia salts are then expelled by very careful heating to a dull red heat, and weighed as potassium and sodium chlorid ($KCl+NaCl$).

The mixed salts are dissolved in water and an excess of platinic chlorid added to the solution. After evaporating nearly to dryness on a steam bath, 20 cc of alcohol is added and allowed to stand one hour. The precipitate is filtered on a weighed Gooch crucible, washed with alcohol, dried at a low heat in an air bath, and weighed as potassium platinic chlorid (K_2PtCl_6), which multiplied by 0.1940 gives potassium chlorid (KCl).

WATER.

For the determination of water, the ordinary method of loss on ignition is not depended upon except in cases in which it is quite certain that carbon dioxid and oxidizable or reducible matter are absent. A sample of about 2 grams of the air-dried powdered material is weighed into a platinum boat, which is placed inside a hard glass combustion tube and strongly ignited in a current of dry air. The expelled water is collected in a sulphuric acid weighing tube, which is connected by a very short piece of rubber tubing to the drawn-out end of the combustion tube.

B.—*Substances mainly soluble in hydrochloric acid.*

In the cases of limestones and dolomites which contain less than 10 per cent of insoluble matter the weighed sample is treated with dilute hydrochloric acid, and the solution, after the filtration of the insoluble residue, is treated exactly as under A, "substances mainly soluble in hydrochloric acid." Whether or not the insoluble residue will require fusion and separate analysis will depend upon the objects of the analysis. Calcareous clays, which are largely insoluble, are best treated as under A, and fused at once.

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[a] Bul. U. S. Geological Survey 176; 97.

II. RATIONAL ANALYSIS.

In the study of clays for various purposes a rational analysis is often called for, and in spite of its great inaccuracy undoubtedly gives results which are of some practical value. The method as given by Meade,[a] which is convenient and rapid, is used with some slight modification in this laboratory.

Heat 1.25 grams of the finely ground sample with 15 cc of concentrated sulphuric acid to near the boiling point of the acid and digest for five hours at this temperature. Cool, dilute, and filter. Wash and ignite the residue to a constant weight, called A. After weighing, brush the residue, which consists of silica, present as sand and undecomposable silicates and silica from the decomposition of the silicates of alumina, into an agate mortar, grind very finely, and weigh 0.5 gram of it into a platinum dish containing 50 cc of boiling caustic potash solution (1.125 sp. gr.). Boil for five minutes, filter, wash first with hot water and then with water containing a little dilute hydrochloric acid, and then again with hot water, dry and ignite to a constant weight. Call this weight B. Multiply A by 0.4 (to correct the 1.25 grams of clay used to correspond to the 0.5 gram of the residue taken for treatment with caustic potash solution) and subtract B from the product. Multiply the difference by 200 to obtain the per cent of silica combined with alumina in the clay. This deducted from the total silica found by analysis gives the silica as sand and undecomposable silicates.

TABLE A.—*Results of laboratory tests.*

[The starred figures are the results obtained by the new method, see page 42.]

Serial No.	Locality.	Name of material.	Coefficient of wear.	Per cent of wear.	Weight in pounds per cubic foot.	Pounds of water absorbed per cubic foot.	Cementing value.
	ALABAMA.						
388	Birmingham, Jefferson County	Chert					*31
389	do	do					*84
391	do	do	3.9	10.2	162.5	1.3	*127
392	do	do	4.9	8.2	159.4	2.2	*113
393	do	do	4.2	9.6	151	8.4	*58
394	do	do					*102
395	do	Blast-furnace slag	5.2	7.6	168.8	.6	[b]137
442	do	Dolomite	7.1	5.7	168.8	1.8	14
443	do	Chert					*390
425	Farmsdale, Hale County	Calcareous clay	2.1	19.1			1,489
426	do	Limestone	2.3	17.4	162.5	2.6	111
390	Tuscaloosa, Tuscaloosa County	Gravel					11
724	Tuskegee, Macon County	do					*400
725	do	do					*75
	ARKANSAS.						
651	Batesville, Independence County	Gravel					17
652	do	do					8
739	Fort Smith, Sebastian County	do					*34
740	do	do					*500
741	do	do					*21
742	do	Sandstone	10	4	156.3	1.4	*90
743	do	do	10.7	3.7	162.5	1.3	*79
438	Hot Springs, Garland County	Chert	3.6	11.1	162.5	.4	*10
	CALIFORNIA.						
578	Los Angeles, Los Angeles County	Granite	2.7	14.8	162.5	.9	4
538	San Jose, Santa Clara County	Chert			156.3	1.8	*13

[a] Examination of Portland Cement. Chem. Pub. Co., 1901.

[b] High cementing value for slag.

TABLE A.—*Results of laboratory tests*—Continued.

[The starred figures are the results obtained by the new method, see page 42.]

Serial No.	Locality.	Name of material.	Coefficient of wear.	Per cent of wear.	Weight in pounds per cubic foot.	Pounds of water absorbed per cubic foot.	Cementing value.
	COLORADO.						
398	Lakeshore, Hinsdale County	Rhyolite	5	8.1	175	4.2	577
399	Capitol City, Hinsdale County	Brecciated felsite	11.9	3.4	156.3	3	12
406	Lakeshore, Hinsdale County	Rhyolite tuff	4.1	9.7	156.3	2.9	513
407	Lake City, Hinsdale County	Andesite	11.5	3.5	162.5	1.9	35
408	do	do	6.3	6.4	162.5	1.9	14
409	do	Diorite (trap)	12.5	3.2	168.8	1	18
396	do	Rhyolite breccia	7	5.7	156.3	2.5	327
397	Sherman, Hinsdale County	Granite	4.6	8.7	162.5	.4	39
	CONNECTICUT.						
354	Ansonia, New Haven County	Basalt	19.2	2.1	189.3	.6	10
349	Branford, New Haven County	Diabase (trap)	11.5	3.5	175.1	.8	49
467	Meriden, New Haven County	do	16.9	2.4	181.3	.7	91
	FLORIDA.						
701	Tampa, Hillsboro County	Calcareous clay	......	......	......	......	*4,000+
702	do	Dolomite	2.2	18.6	162.5	4.8	*94
	GEORGIA.						
422	Atlanta, Fulton County	Hornblende gneiss	10.2	4	187.5	.1	3
419	Augusta, Richmond County	Gneiss	15.7	2.6	162.5	.3	8
417	Bartow County	Dolomite	5	8	178.1	.7	17
583	Canton, Cherokee County	Eclogite	13.8	2.9	231.3	.1	3
424	Chickamauga, Catoosa County	Limestone	7.1	5.7	168.8	.1	99
415	Coweta County	Olivine diabase (trap).	17.3	2.3	193.8	.2	2
421	Lithonia, Dekalb County	Granite	8.3	4.8	168.8	.1	2
416	New Elberton, Elbert County	do	13.1	3	168.8	.1	2
420	Roberts Station, Jones County	Olivine diabase (trap).	15.2	2.6	187.5	.3	6
698	Savannah, Chatham County	Gravel	......	......	......	......	*233
699	do	do	......	......	......	......	*176
423	Stone Mountain Station, Dekalb County	Granite	7.5	5.3	162.5	.1	13
468	Toccoa, Habersham County	Gneiss	7.7	5.2	162.5	.2	2
413	Walker County	Limestone	10.6	3.8	162.5	3.5	74
414	do	Chert	1.4	27.9	143.7	3.3	*12
418	do	do	2.5	16.4	184.4	3.7	*35
	ILLINOIS.						
716	Chicago, Cook County	Dolomite	9.1	4.4	168.8	.6	*18
756	do	do	6.9	5.8	168.8	.8	*28
451	Dixon, Lee County	Dolomite gravel	......	......	......	......	9
437	Elco, Alexander County	Novaculite (chert)	6.8	5.9	153.6	3.3	72
584	do	do	14.6	2.7	151.0	4.1	*15
465	Forreston, Ogle County	Gravel	......	......	......	......	14
454	Kenney, Dewitt County	do	......	......	......	......	16
452	Menominee, Jo Daviess County	Limestone	8	13.1	168.8	1.8	27
450	Ullin, Pulaski County	do	7.2	5.6	156.3	3.3	127
	INDIANA.						
540	Bicknell, Knox County	Dolomitic limestone.	10.1	4	168.8	.9	23
515	Freelandville, Sullivan County	Limestone	10.6	3.8	171.8	.7	38
588	do	Dolomite	16.1	2.5	175	1.3	36
456	Merom, Sullivan County	Gravel	......	......	......	......	10
719	North Vernon, Posey County	Dolomite	......	......	162.5	2.2	......
516	Spencer, Owen County	Limestone	13	3.1	168.8	.9	166
	IOWA.						
383	Butterville, Tama County	Limestone	1.8	21.9	162.2	3.2	22
557	Mason City, Cerrogordo County	Clay	......	......	......	......	250
438	Montour, Tama County	Limestone	5.9	6.8	163.1	1.7	129
455	Peosta, Dubuque County	Dolomite	3.4	11.6	156.3	3.3	32
462	Raymond, Blackhawk County	do	8.9	4.6	168.8	.7	161

TABLE A.—*Results of laboratory tests*—Continued.

[The starred figures are the results obtained by the new method, see page 42.]

Serial No.	Locality.	Name of material.	Coefficient of wear.	Per cent of wear.	Weight in pounds per cubic foot.	Pounds of water absorbed per cubic foot.	Cementing value.
	KENTUCKY.						
446	Lexington, Fayette County	Limestone	6.4	6.2	168.8	.8	58
447	do	do	11.9	3.4	162.5	3.5	56
457	Kuttawa, Lyon County	Sandstone	2.2	17.8	151	8.2	55
461	Cedar Bluff, Caldwell County	Limestone	8.9	4.5	140.7	3.1	80
	MARYLAND.						
759	Baltimore, Baltimore City	Clay					*60
760	do	do					*35
580	Fort Washington, Prince George County.	Gravel					2
487	Frederick, Frederick County	Limestone	6.6	6	168.8	.1	26
427	Cumberland, Allegany County	Sandstone	4.9	8.2	125	9.4	44
428	do	Calcareous shale	2.5	16.2	168.8	1.9	380
429	do	Limestone	3.4	11.7	165.6	1.6	133
430	do	do	3	13.4	168.8	1.7	143
363	Dickerson, Montgomery County	Diabase (trap)	14.2	2.8	186.6	.1	60
343	Frederick, Frederick County	Dolomite	8.9	4.5	176.6	.3	32
439	do	Limestone	6.8	5.9	163	.2	33
	MASSACHUSETTS.						
367	Brookline, Norfolk County	Metamorphic slate.	8.5	4.7	175.6	.5	151
368	do	Rhyolite	22.9	1.8	178.1	.06	24
369	do	Diabase	18.9	2.1	179.6	.4	9
718	Quincy, Norfolk County.	Chlorite schist	7.9	5.1	181.3	.1	*28
611	Salem, Essex County	Augite diorite (trap).	14.7	2.7	187.5	.3	18
444	Taunton, Bristol County	Gravel			168.8	.5	13
445	do	Granite	9.7	4.1	168.8	1.5	13
411	Webster, Worcester County	Schist	6.4	6.3	175	.3	18
517	Westfield, Hampden County	Diabase (trap)	30.7	1.3	187.5	.5	76
386	Winchester, Middlesex County	do	6.4	6.3	200.6	.8	21
	MISSISSIPPI.						
460	Brookhaven, Lincoln County	Gravel					16
464	Sardis, Panola County	do					58
453	Stonington, Jefferson County	Sandstone	1.3	31.8	131.2	11.6	53
	MISSOURI.						
351	Gumbo, St. Louis County	Gravel					12
	NEBRASKA.						
360	Bluesprings, Gage County	Chert	6.9	5.8	160.6	1.3	*102
	NEW HAMPSHIRE.						
370	Hanover, Grafton County	Granite	12.6	3.2	165.6	.2	3
371	do	Schist	12.3	3.3	194.1	.2	32
372	Lebanon, Grafton County	Hornblende schist	9.9	4	189.7	.4	25
373	do	Biotite schist	6.7	5.9	169.1	.5	49
	NEW JERSEY.						
357	Chimney Rock, Somerset County	Basalt	23.6	1.7	186.3	.1	72
720	Gladstone, Somerset County	Shale			156.3	4.8	*985
387	Lambertville, Hunterdon County	Gabbro (trap)	14.3	2.8	183.7	.1	131
405	Middle Valley, Hunterdon County	Diabase (trap)	23.8	1.7	187.5	.1	19
341	Millville, Cumberland County	Gravel					1,680
350	New Providence, Union County	Diabase (trap)	23.6	1.7	186.3	.4	19
478	Wanaque, Passaic County	Peridotite	11.1	3.6	209.4	.3	3
566	Shady Side Quarries	Diabase (trap)	19.7	2	184.4	.5	38
637	Somerville, Somerset County	Basalt tuff	2.4	16.6	147.5	6.4	6
669	do	do	4.3	9.3	151	5.7	14

TABLE A.—*Results of laboratory tests*—Continued.

[The starred figures are the results obtained by the new method, see page 42.]

Serial No.	Locality.	Name of material.	Coefficient of wear.	Per cent of wear.	Weight in pounds per cubic foot.	Pounds of water absorbed per cubic foot.	Cementing value.
	NEW YORK.						
404	Bellona, Yates County	Limestone	7.5	5.3	168.8	.1	55
356	Brockport, Monroe County	do	8.9	4.5	169.4	.3	9
400	Canandaigua, Ontario County	do	8.8	4.6	168.8	.2	54
344	Deerpark, Orange County	Sandstone					54
382	do	Shale	8.6	4.7	166.3	1.4	62
346	Geneva, Ontario County	Limestone	13.9	2.9	164.1	1.2	8
347	do	do	8.8	4.5	170.9	.2	14
340	Hudson, Columbia County	do	7.6	5.3	169.7	.5	95
385	do	Slate	5.8	6.9	169.4	.4	202
410	Leroy, Genesee County	Flint	5.5	7.2	162.5	.2	31
532	Littlefalls, Herkimer County	Gneiss	20.7	1.9	168.8	.3	2
732	North Leroy, Genesee County	Limestone	8.5	4.7	168.8	.1	*138
733	do	do	7.9	5.1	162.5	.4	*74
335	Rockland Lake, Rockland County	Diabase (trap)	13.2	3	192.5	.5	110
470	Tomkins Cove, Rockland County	Dolomite	6.7	5.9	168.8	.3	*80
	NORTH CAROLINA.						
514	Ashboro, Randolph County	Felsite	21.3	1.9	168.8	.02	2
469	Asheville, Buncombe County	Hornblende schist	7.1	5.7	200	.2	6
627	do	Granite	18.6	2.2	168.8	.2	6
628	do	Diorite	17.4	2.3	181.3	.2	1
705	do	Hornblende schist	11	3.6	193.8	.5	*7
434	Concord, Cabarrus County	Granite	21.7	1.8	162.5	.3	3
435	do	Mixed stone	4.6	8.7			12
436	do	Granite	7	5.7	168.8	.2	2
513	do	Syenite	14.4	2.8	168.8	.3	2
431	Elm City, Wilson County	Granite	14.2	2.8	165.6	.4	8
539	Fayetteville, Cumberland County	Gravel					43
504	Hot Springs, Madison County	Dolomite	9.5	4.2	181.3	.1	2
535	Morganton, Burke County	Basalt	20.3	2	187.5	.2	2
380	Newbern, Craven County	Clay					45
381	do	Limestone	1.2	34.2	138.1	1.9	79
366	Pinehurst, Moore County	Gravel					24
432	Wilson, Wilson County	Granite	6.2	6.4	162.5	.4	9
433	do	do	7.2	5.6	159.4	.2	20
401	Wilmington, New Hanover County	Conglomerate	3.2	12.7	156.3	2.5	125
402	do	Limestone	4	9.9	147.4	2.7	127
403	do	do					110
	OHIO.						
663	Columbus, Franklin County	Limestone	6	6.7	159.4	3.2	12
664	do	do	5.8	6.9	162.5	1.5	41
666	do	do	10.6	3.8	156.3	2.6	58
665	do	Dolomite	7	5.7	159.4	2.7	10
582	East Liberty, Stone County	Dolomitic limestone.	10.6	3.8	175	.8	28
707	Kirtland, Lake County	Fieldstone (erratics.)	8	5			*7
345	Lorain, Lorain County	Blast-furnace slag	4.8	8.2	140.3	3.6	19
570	Medina, Medina County	Gravel					4
	PENNSYLVANIA.						
339	Cedar Hollow, Chester County	Dolomite	10.6	3.8	178.1	.2	2
365	Hatboro, Montgomery County	Sandstone	10	4	166.9	.8	323
352	Northampton County	Diorite (trap)	14.3	2.8	182.2	.3	5
730	Redington, Northampton County	Dolomite	7.3	5.5	181.3	.1	*36
731	Rockhill, Huntingdon County	Diabase (trap)	34.5	1.2	193.8	.1	*30
697	Wilkesbarre, Luzerne County	Sandstone	11.6	3.4	168.8	.5	*202
	RHODE ISLAND.						
659	Providence, Providence County	Augite diorite (trap.)	20.6	1.9	187.5	.2	19
	SOUTH CAROLINA.						
488	Barnwell County	Gravel					50
374	Columbia, Richland County	Granite	13.9	2.9	163.8	.2	5
375	Pacolet, Spartanburg County	do	20.9	1.9	166.8	.2	7

TABLE A.—*Results of laboratory tests*—Continued.

[The starred figures are the results obtained by the new method, see page 42.]

Serial No.	Locality.	Name of material.	Coefficient of wear.	Per cent of wear.	Weight in pounds per cubic foot.	Pounds of water absorbed per cubic foot.	Cementing value.
	SOUTH DAKOTA.						
449	Rowena, Minnehaha County	Quartzite	15.1	3.7	168.8	0.1	2
	TENNESSEE.						
440	Bartlett, Shelby County	Gravel					174
448	Chattanooga, Hamilton County	Limestone	14.2	2.8	168.8	.3	47
503	do	do	10.1	4	168.8	2.1	54
342	Columbia, Maury County	Chert					1,083
336	Graytown, Hickman County	Limestone	8.3	12	167.5	4.8	158
337	do	do	5.9	6.8	174.4	1.7	59
338	do	Chert	8.8	4.4	155.8	1.7	*240
359	do	Limestone	5.1	7.9	192.8	3.2	114
361	do	do	5.2	7.8	176.3	2.2	158
441	Memphis, Shelby County	Gravel					249
376	Nashville, Davidson County	Limestone	8	5	169.4	.2	231
377	do	do	7.7	5.2	168.4	.2	195
378	do	do	6.7	6	167	1.8	87
379	do	do	2.7	15.1	156.3	3.1	63
	TEXAS.						
536	Austin, Travis County	Limestone gravel					77
	VERMONT.						
726	Wallingford, Rutland County	Amphibolite	8.5	4.6	193.8	.3	*16
	VIRGINIA.						
612	Albemarle County	Chlorite schist	10.1	4	187.5	.2	8
613	do	Gneiss	9.8	4.1	168.8	.1	1
412	Alexandria County	Mica schist	9.3	4.3	173.8	.3	13
493	Basic City, Augusta County	Quartzite			159.4	1.3	2
537	Blacksburg, Montgomery County	Dolomite	11.2	3.6	181.3	.3	10
531	Burrowsville, Prince George County	Clay					2,531
364	Charlottesville, Albemarle County	Amphibolite	9.9	4.1	183.8	.4	86
489	do	Limestone	7	5.7	168.8	.2	13
490	do	Diorite	12.8	3.1	200	.8	5
491	do	Chlorite schist	5.7	7.1	184.4	.3	9
494 495	do	Uralite diabase (trap).	17.3	2.3	181.3	.6	25
496	do	Hornblende schist	20.6	1.9	181.3	.6	9
497	do	do	9.7	4.1	187.5	1.1	7
498	do	Biotite schist	11.7	3.4	165.6	.3	2
533	do	Clay					1,661
534	do	do					475
548	do	Amphibolite (trap).	19.5	2.1	187.5	.6	5
549	do	do	26.7	1.5	190.6	.4	3
550	do	do	18.3	2.2	193.8	.2	6
551	do	do	24	1.7	193.8	.5	6
552	do	Hornblende schist	16.2	2.5	181.3	.9	10
500	Shadwell, Albemarle County	Chlorite schist	11.3	3.5	187.5	.3	14
492	Craigsville, Craig County	Limestone	9.7	4.1	168.8	.3	22
750	Fredericksburg, Spottsylvania County	do	7.3	5.5	175	1.5	*15
721	Jackson City, Alexandria County	Yellow clay					*500
722	do	Blue clay					*424
723	do	Yellow clay					*1,500
362	Lawrenceville, Brunswick County	Clay					3,059
499	Monticello Mountain, Albemarle County	Quartzite	11.1	3.6	162.5	.4	4
581	Occoquan, Prince William County	Gneiss	11.7	3.4	175	.3	2
717	Manassas, Prince William County	Sandstone					*8
	WISCONSIN.						
667	Berlin, Green Lake County	Rhyolite	22.5	1.8	168.8	.05	2
668	do	do	10.1	4	168.8	.03	*1
746	Madison, Green Lake County	do	17	2.3	162.5	.08	*8
695	Utley, Green Lake County	do	8	5	162.5	.04	*5
696	do	do	7.2	5.6	162.5	.06	*5
747	do	Dolomite	9.8	4.1	168.8	.8	*60

TABLE A.—*Results of laboratory tests*—Continued.

[The starred figures are the results obtained by the new method, see page 42.]

Serial No.	Locality.	Name of material.	Coefficient of wear.	Per cent of wear.	Weight in pounds per cubic foot.	Pounds of water absorbed per cubic foot.	Cementing value.
	WISCONSIN—Continued.						
734	Tomah, Monroe County	Gravel					*725
735	do	do					*295
736	do	do					*197
	WASHINGTON.						
476	Seattle, King County	Quartzite	18.2	2.2	168.8	.6	47
477	do	Basalt (trap)	19.8	2	181.3	1.9	25
508	do	Chlorite schist	11	3.6	168.8	1	272
	DISTRICT OF COLUMBIA.						
510	Washington	Shell marl					58
511	do	do					741
512	do	do					931
	CUBA.						
472	Campo Florida	Diorite (trap)	11.7	3.4	175.0	.7	137
471	Cienfuegos	Andesite (trap)	15.2	2.6	168.8	2.2	337
473	Habana	Limestone	10.1	4	165.6	1	90
575	do	Diorite	18.3	2.2	168.8	.8	8

TABLE B (PART I).—*Percentage of common mineral constituents*

[The small numerals placed above the percentage figures denote, respectively, whether the mineral posed, and the secondary those resulting from subsequent change. In some cases the separate deter- two is given in one column and an asterisk or dagger indicates the other mineral.]

	Locality.	Serial No.	Scientific name.	Common name.	Orthoclase.	Plagioclase.	Quartz.	Hornblende.
1	Charlottesville, Va	364	Amphibolite	Trap			$^{1}5$	$^{1}51$
2	Do	548	do	do		$^{1}3$	$^{1}2$	$^{1}87$
3	Do	549	do	do			$^{1}4$	$^{1}85.8$
4	Do	550	do	do			$^{1}4$	$^{1}82$
5	Do	551	do	do		$^{1}1.5$	$^{1}2.5$	$^{1}80$
6	Wallingford, Vt	726	do	do	$^{1}21$		$^{1}7$	$^{1}61.8$
7	Cienfuegos, Cuba	471	Andesite	do		$^{1}40$	$^{2}15$	
8	Lake City, Colo	407	do	do		$^{1}20$	$^{2}30$	
9	Do	408	do	do		$^{1}55$		
10	Ansonia, Conn	354	Basalt	do		$^{1}24.4$		
11	Boundbrook, N. J	357	do	do		$^{1}23$		
12	Do	252	do	do		$^{1}38$		
13	Byram Station, N. J	251	do	do		$^{1}42$		
14	Deerfield, Franklin County, Mass.	268	do	do		$^{1}35$		
15	Guttenberg, N. J	73	do	do		$^{1}50$		
16	Morganton, N. C	535	do	do		$^{1}23$		
17	Seattle, Wash	477	do	do		$^{1}30$		
18	Great Notch, N. J	260	do	do		$^{1}21$		
19	Somerville, N. J	637	Basalt-tuff	do	$^{1}3$			
20	Do	669	do	do	$^{1}3.6$			
21	Wilmington, N. J	250	Basalt	do		$^{1}30$		
22	Salisbury, Essex County, Mass.	33	Camptonite	do		$^{1}60$		$^{1}25$
23	Birmingham, Ala	391	Chert	Chert			$^{1-2}99$	
24	Do	392	do	do			$^{1}99$	
25	Do	393	do	do			$^{1}99$	
26	Bluesprings, Nebr	360	do	do			$^{1}94$	
27	Elco, Ill	437	do	do			$^{1}97.9$	
28	Do	584	do	do			$^{1}96.5$	
29	Graytown, Tenn	338	do	do	$^{1}0.7$		$^{1}88.4$	
30	Hot Springs, Ark	438	do	do			$^{1}99.8$	
31	San Jose, Cal	538	do	do			$^{1}88.3$	
32	Amherst, Hampshire County, Mass.	78	Diabase	Trap		$^{1}50$		
33	Do	82	do	do		$^{1}40$		
34	Brookline, Norfolk County, Mass.	7	do	do		$^{1}40$		
35	Do	369	Diabase (altered)	do		$^{1}32.5$	$^{2}4$	$^{2}2.4$
36	Do	23	Diabase	do		$^{1}45$	$^{2}10$	
37	Charlottesville, Va	494	Diabase (uralitic)	do		$^{1}32$	$^{1}3$	$^{2}50$
38	Coweta County, Ga	415	Diabase	do		$^{1}42.2$		
39	Deerfield, Franklin County, Mass.	76	do	do		$^{1}65$		
40	Dickerson, Md	363	do	do		$^{1}33$		$^{2}1$
41	Everett, Middlesex County, Mass.	2	do	do		$^{1}40$	$^{2}3$	
42	Great Notch, N. J	261	do	do		$^{1}31$		
43	Haverstraw, N. Y	49	do	do		$^{1}50$		
44	Holyoke, Hampden County, Mass.	66	do	do		$^{1}50$		
45	Hydepark, Norfolk County, Mass.	9	do	do		$^{1}35$		
46	Ipswich, Essex County, Mass.	61	Diabase (uralitic)	do		$^{1}18$		$^{1}66$
47	Lynn, Essex County, Mass	21	Diabase	do		$^{1}40$		
48	Do	29	do	do		$^{1}40$		
49	Malden, Middlesex County, Mass.	270	do	do		$^{1}34.2$		
50	Medford, Middlesex County, Mass.	20	do	do		$^{1}50$	$^{1}3$	
51	Meriden, Conn	11	do	do		$^{1}50$		
52	Do	71	do	do		$^{1}43$		
53	Do	467	do	do		$^{1}25$		
54	Middle Valley, N. J	405	do	do		$^{1}29.3$	$^{1}2$	
55	Milton, Norfolk County, Mass.	219	Diabase (uralitic)	do		$^{1}25$		$^{2*}57$
56	Monson, Hampden County, Mass.	68	Diabase	do		$^{1}55$		
57	Newbury, Essex County, Mass.	31	do	do		$^{1}60$		
58	New Providence, N. J	350	do	do		$^{1}20$		
59	Newton, Middlesex County, Mass.	6	do	do		$^{1}50$	$^{2}5$	

in road-making rocks (*see Part II for completed analysis*).

is primary or secondary, the primary minerals being the original minerals of which the rock is com-
mination of two minerals in the same rock is impracticable, therefore the total percentage of the

Augite.	Magnetite.	Apatite.	Calcite.	Olivine.	Biotite.	Chlorite.	Serpentine.	Epidote.	Pyrite.	Muscovite.	Microcline.	Garnet.	Titanite.	
	^{1}3							^{1}41						1
					^{1}0.6			^{1}7.3						2
	^{1}0.1							^{1}10	^{1}0.1					3
	^{1}3.5							^{1}10	^{1}0.4					4
					^{1}3			^{1}6					^{1}7	5
	^{1}6		^{2}1		^{2}0.1		^{2}2		^{1}0.1			^{1}1		6
	^{1}5	^{1}1	^{2}2			^{2*}22		(^{2*})						7
	^{1}4		^{2}4.5			^{2}10		^{2}25		^{2}5.0				8
^{1}43	^{1}2													9
^{1}71.5	^{1}3.5			^{1}0.4			^{2}0.2							10
^{1}59	^{1}3						^{2}5							11
^{1}27	^{1}4					^{2}5								12
^{1}54	^{1}3				^{2}1									13
^{1}30	^{1}3.9					^{2}4			^{2}0.1					14
^{1}40	^{1}10													15
^{1}58	^{1}2			^{1}15	^{2}0.1		^{2}1.9							16
^{1}40	^{1}10						^{2}20							17
^{1}50							^{2}5							18
	^{1}2.4		^{2}17.2		^{1}0.4									19
	^{1}3		^{2}9.2		^{1}0.5									20
^{1}30	^{1}3					^{2}2								21
	$^{1-2\dagger}$5					^{2}10							($^{1-2\dagger}$)	22
	^{1}0.3					^{2}0.2								23
	^{1}0.1								^{1}0.1	^{2}0.3				24
	^{1}0.1								^{1}0.1	^{2}0.3				25
									^{1}0.1	^{1}0.9		^{1}1.2		26
	^{1}0.1													27
	^{1}0.1									^{1}0.1				28
	^{1}0.4				^{1}0.1					^{2}0.7		^{1}6.7		29
	^{1}0.1													30
			^{2}2.4											31
^{1}40	$^{1-2}$5					^{2}5								32
^{1}50	^{1}10													33
	$^{1-2}$15					^{2}40			^{1}5					34
	^{1}0.2					^{2}0.4		^{2}57.3						35
			^{2}10			^{2*}35	(^{2*})							36
	^{1}5							^{2}5	^{2}5					37
^{1}51	^{1}5.5					^{2}0.1			^{2}0.2	^{2}1				38
	$^{1-2}$10			^{1}20		^{2*}5	(^{2*})							39
^{1}55	^{1}4.8	^{1}0.1	^{2}1		^{1}0.1	^{2}1				^{2}4				40
	$^{1-2}$10	^{1}2		^{1}15	^{1}20	^{2*}10	(^{2*})							41
^{1}37	^{1}5						^{2}5							42
^{1}40	$^{1\ 2}$5						^{2}5							43
^{1}40	^{1*}10								(^{1*})					44
^{1}30	^{1}10	^{1}3			^{1}12	^{2†}10		(^{2†})						45
	^{1}5							^{2}11						46
^{1}40	$^{1-2}$8					^{2†}12			(^{2†})					47
^{1}30	$^{1-2}$10					^{2}20								48
^{1}19	^{1}7		^{2}2.3			^{2}9		^{2}0.5						49
	^{2}10			^{1}5		^{2†}30	(^{2†})	(^{2†})	^{2}2					50
^{1}10	$^{1-2}$10		^{2}5			^{2†}25	(^{2†})							51
^{1}17	^{1}7		^{2}10			^{2}11								52
^{1}40	^{1}4		^{2}5			^{2}10				^{2}10				53
^{1}30	^{1}8	^{1}0.5			^{1}1	^{2}9			^{1}0.2					54
	^{1}5				^{2}13	(^{2*})								55
^{1}40	^{1*}5								(^{1*})					56
^{1*}35	$^{1-2}$2			(^{1*})		^{2}3								57
^{1}30	^{1}4					^{2}5								58
	$^{1-2}$10					^{2}25		^{2}10						59

TABLE B (PART I).—*Percentage of common mineral constituents in*

	Locality.	Serial No.	Scientific name.	Common name.	Orthoclase.	Plagioclase.	Quartz.	Hornblende.
60	Quincy, Norfolk County, Mass.	18	Diabase	Trap		[1]*50		
61	Do	299	Diabase (uralitic)	do			(2†)	[2]†79.4
62	Roberts Station, Ga	420	Diabase	do		[1]58.3		
63	Rockhill, Pa	731	do	do		[1]63.6		
64	Rockland Lake, N. Y	335	do	do	[1]1	[1]40	[1]1	[2]5
65	Saugus, Essex County, Mass	22	do	do		[1]45		
66	Do	25	do	do		[1]40		
67	Do	28	do	do		[1]40		
68	Do	32	do	do		[1]50		
69	Do	84	do	do		[1]50		
70	Shadyside, N. J	566	do	do		[1]49		[2]11
71	Somerville, Middlesex County, Mass.	19	Diabase (gabbroitic).	do		[1]56.4		
72	Do	75	Diabase	do		[1]45		
73	Sterling, Worcester County, Mass.	83	do	do		[1]55.3		
74	Ware, Hampshire County, Mass.	64	do	do		[1]35		
75	Westfield, Hampden County, Mass.	517	do	do		[1]48		
76	West Springfield, Hampden County, Mass.	12	do	do		[1]45		
77	Do	67	do	do		[1]40		
78	Do	91	do	do		[1]50		
79	Do	93	do	do		[1]50		
80	Winchester, Middlesex County, Mass.	59	do	do		[1]30		
81	Asheville, N. C	628	Diorite	do	[1]25	[1]6	[1]24.9	[1]35
82	Beverly, Essex County, Mass.	117	do	do	[1]27	[1]6	[1]5	[1]33
83	Campo, Fla	472	do	do		[1]60	[1]15	[1]15
84	Charlottesville, Va	490	do	do		[1]22		[1]72
85	Delaware River, Pa	352	do	do		[1]44.8	[1]9	[1]16
86	Havana, Cuba	575	do	do		[1]4	[1]5	[1]27
87	Lake City, Colo	409	do	do		[1]67.5	[1]7	
88	Millville, Worcester County, Mass.	227	do	do	[1]10	[1]31.3	[1]9	[1]30
89	Newbury, Essex County, Mass.	35	do	do	[1]*40	(1*)	[1]10	[1]40
90	Providence, R. I	659	Augite-diorite	do		[1]33		[1]37.2
91	Salem, Essex County, Mass.	1	do	do	[1]*50	(1*)		[1]20
92	Do	611	Diorite (vogesite)	do	[1]63	[1]3		[1]1
93	Watertown, Middlesex County, Mass.	96	Diorite	do		[1]55	[2]2	[1]15
94	Winchester, Middlesex County, Mass.	56	do	do		[1]40	[2]2	[1]45
95	Do	304	do	do		[1]46		[1]34
96	Canton, Ga	583	Eclogite	Garnet rock			[1]21	[1]3.3
97	Glen Mills, Pa	254	do	do		[1]40		[1]1
98	Ashboro, N. C	514	Felsite	Porphyry	[1]*81.7		(1*)	
99	Boston, Suffolk County, Mass.	40	do	do	[1]*90	(1*)	[1]8	
100	Lynn, Essex County, Mass.	24	do	do	[1]*95		(1*)	
101	Do	27	do	do	[1]*85	(1*)	[1]5	
102	Quincy, Norfolk County, Mass.	72	do	do	[1]*80		[1]5	
103	Revere, Suffolk County, Mass.	3	do	do	[1]95		[1]2	
104	Do	124	do	do	[1]*97.4		(1*)	
105	Rowley, Essex County, Mass.	97	do	do	[1]*95		(1*)	
106	Saugus, Essex County, Mass.	86	do	do	[1]*90		(1*)	
107	Do	87	do	do	[1]*95	(1*)	[1]†5	
108	Weymouth, Norfolk County, Mass.	92	do	do	[1]*90		(1*)	
109	Bergen, N. J	249	Gabbro	Trap		[1]44		[2]0.5
110	Cortland, N. Y	195	do	do		[1]48		
111	Lambertsville, N. J	253	Uralite-gabbro	do		[1]23	[1]3	[2]26
112	Do	387	Gabbro	do		[1]35		[2]17
113	Rocky Hill, N. J	218	do	do		[1]68		
114	Do	285	Uralite-gabbro	do		[1]9	[1]8	[1-2]27
115	Athol, Worcester County, Mass.	88	Gneiss	Gneiss	[1]8	[1]0.2	[1]80.8	
116	Albemarle County, Va	613	do	do	[1]26.4	[1]7	[1]45	
117	Atlanta, Ga	422	Hornblende-gneiss	do		[1]4.2	[1]23.5	[1]70
118	Auburn, Worcester County, Mass.	203	Gneiss	do	[1]51.4	[1]3	[1]30	

road-making rocks (see Part II for completed analysis)—Continued.

Augite.	Magnetite.	Apatite.	Calcite.	Olivine.	Biotite.	Chlorite.	Serpentine.	Epidote.	Pyrite.	Muscovite.	Microcline.	Garnet.	Titanite.	
	^{2}5		^{2}10			^{2}30		^{2}5		(^{1*})				60
^{1}9	^{1}7				^{2}0.1	^{2}4.5		(^{2}†)						61
^{1}30	^{1}6.2					^{2}1.1								62
^{1}35	^{1}.2				^{1}0.8	^{2}0.4								63
^{1}35	^{1}9.9	^{1}0.1				^{2}1								64
^{1}35	$^{1-2}$10					^{2*}10	(^{2*})							65
^{1}30	$^{1-2}$10				^{1}2	^{2*}10	(^{2*})	^{2}5					^{1}3	66
^{1}20	$^{1-2}$15	^{1*}2					^{2}†23	(^{2}†)					(^{1*})	67
^{1}25	$^{1-2}$8					^{2*}15		(^{2*})					^{1}2	68
	^{1}5			^{1}20		^{2*}20	(^{2*})	^{2}5						69
^{1}22	^{1}2.5				^{1}0.5					^{2}9				70
^{1}12	^{1}2	^{1}0.5	^{2}3		^{1}2	^{2}10		^{2}0.1						71
	^{1}10			^{1*}25	^{1}15		(^{2*})							72
^{1}41	^{1}3		^{2}0.2		^{1}0.3		^{2}0.1							73
^{1}60	^{1}5													74
^{1}39	^{1}3			^{1}4		^{2}1	^{2}2							75
^{1}50	$^{1-2*}$5					(^{2*})								76
^{1}50	^{1*}10								(^{1*})					77
^{1}30	^{1}5					^{2}15								78
^{1}30	^{1}10					^{2}10								79
^{1}50						^{2}15			$^{1-2}$5					80
		^{1}1							^{1}4			^{1}2	^{1}1	81
^{1}1	^{1}2	^{1}1	^{2}1		^{1}8									82
	^{1*}1	(^{1*})				^{2}2			^{2}5					83
	^{1}2												^{2}4	84
	^{1}5				^{1}7			^{2}8						85
	^{1}0.2		^{2}4			^{2}2								86
	^{1}6	^{1}0.1	^{2}4		^{1}13	^{2}2		^{2}0.2					^{1}0.1	87
	^{1}5	^{1}0.1	^{2}2		^{1}6			^{2}4	^{1}1					88
	^{1}2	^{1}1	^{2*}4			(^{2*})							^{1}3	89
^{1}5	^{1}6	^{1}0.5	^{2}0.1		^{1}7			^{2}0.1	^{1}0.1					90
^{1}16	^{1}10	^{1}†4	(^{2}†)											91
^{1}15.8	^{1}8	^{1}1				^{2}7.2								92
	^{1*}5		^{2}3		^{1}10	^{2}†10		(^{2}†)	(^{1*})					93
	^{1*}10	^{1}3							(^{1*})					94
	^{1}2	^{1}0.3			^{1}2.5			^{2}0.1	^{2}0.1					95
	^{1}1.3		^{2}1			^{2}3						^{1}70.4		96
^{1}27	^{1}6		^{2}1									^{1}25		97
	^{1}0.1	^{1}0.1			^{1}10	^{2}†8.1		(^{2}†)						98
									^{1}2					99
	^{1}5													100
														101
	^{1}5					^{2}10				(^{2*})				102
						^{2}3								103
	^{1}0.1		^{2}0.1			^{2}0.2		^{2}0.1						104
	^{1}5													105
	$^{1-2}$†10					(^{2}†)		(^{2}†)						106
					(^{2}†)									107
	^{1}†10					(^{2}†)								108
^{1}42.5	^{1}2	^{1}1			^{1}2	^{2}1								109
^{1}13	^{1}2	^{1}1			^{1}22									110
^{1}15	^{1}2				^{1}1									111
^{1}28	^{1}1				^{1}0.7	^{2}0.1								112
^{1}24	^{1}2				^{1}2	^{2}1								113
^{1}8	^{1}1	^{1}0.1	^{2}0.1		^{1}1			^{2}10						114
	^{1}0.4	^{1}0.1			^{1}10				^{2}0.1				^{1}0.2	115
	^{1}0.1				^{1}1.4					$^{1-2}$19				116
		^{1}0.3							^{1}2					117
	^{1}0.5				^{1}4					^{1}10	^{1}0.5	^{1}0.4		118

TABLE B (PART I).—*Percentage of common mineral constituents in*

	Locality.	Serial No.	Scientific name.	Common name.	Orthoclase.	Plagioclase.	Quartz.	Hornblende.
119	Augusta, Ga	419	Granite-gneiss	Gneiss	[1]27	......	[1]60.8	......
120	Ashby, Middlesex County, Mass.	118	Gneiss	do	[1]49	[1]7	[1]27	......
121	Dartmouth, Bristol County, Mass.	77	do	do	[1]*55[a]	......	[1]30	......
122	Duxbury, Plymouth County, Mass.	5	do	do	[1]*40	([1]*)	[1–2]50	......
123	Lee, Berkshire County, Mass.	51	Hornblende gneiss	do	[1]14	[1]2	[1]50	[1]14
124	Little Falls, N. Y	532	Granite gneiss	do	[1]40	......	[1]49	[1]9
125	Marion, Plymouth County, Mass.	13	Gneiss	do	[1]30	......	[1–2]30	......
126	Newburyport, Essex County, Mass.	37	do	do	[1]*40	([1]*)	[1]40	......
127	Occoquan, Va	581	do	do	[1]18	[1].1	[1]53.4	......
128	Round Island, N. Y	100	do	do	[1]15	......	[1]15	[1]10
129	Toccoa, Ga	468	Granite gneiss	do	......	......	[1]50	......
130	Uxbridge, Worcester County, Mass.	63	Gneiss	do	[1]30	[1]20	[1]10	[1]35
131	Westport, Bristol County, Mass.	81	do	do	[1]45	......	[1]*40	......
132	Ashby, Middlesex County, Mass.	60	Granite	Granite	[1]25	[1]7	[1]56.9	......
133	Asheville, N. C	627	do	do	[1]55	......	[1]25	......
134	Beverly, Essex County, Mass.	62	Hornblende granite	do	[1]50	[1]10	[1]20	[1]8
135	Do	74	Granite	do	[1]40	......	[1]20	......
136	Do	89	Hornblende granite	do	[1]40	[1]10	[1]30	[1]10
137	Clinton, Worcester County, Mass.	242	Gneissoid granite	do	[1]8	......	[1]66	......
138	Do	279	Granite	do	[1]12	[1].3	[1]66.6	......
139	Columbia, S. C	374	do	do	[1]43.4	[1]1	[1]31	[1].4
140	Concord, N. C	434	do	do	[1]8	[1]5	[1]70	......
141	Do	436	do	do	[1]25	......	[1]5	......
142	Elberton, Ga	416	do	do	[1]77.3	[1]3	[1]9	......
143	Elm City, N. C	431	do	do	[1]36.5	[1]10	[1]30	......
144	Fitchburg, Worcester County, Mass.	256	do	do	[1]52.5	[1]2	[1]30	......
145	Gloucester, Essex County, Mass.	26	Hornblende granitite	do	[1]53.8	[1]2	[1]15	[1]11
146	Hanover, N. H	370	Gneissoid granite	do	[1]29.7	[1].2	[1]30	......
147	Lithonia, Ga	421	Granite	do	[1]32	[1]6	[1]41.7	......
148	Los Angeles, Cal	578	do	do	[1]53	[1]8.6	[1]32	......
149	Northampton, Hampshire County, Mass.	8	Hornblende granitite	do	[1]33.2	[1]2	[1]22	[1]11
150	Orange, Franklin County, Mass.	39	do	do	[1]15	[1]33	[1]40.6	[1].4
151	Pacolet, S. C	375	Granite	do	[1]48	......	[1]36.6	......
152	Quincy, Norfolk County, Mass.	17	do	do	[1]†75[a]	......	[1]20	[1]*5
153	Rockport, Essex County, Mass.	34	do	do	[1]*75	([1]*)	[1]23	......
154	Saugus, Essex County, Mass.	15	do	do	[1]*30	([1]*)	[1]55	......
155	Do	32	do	do	[1]35	[1]10	[1]50	[1]5
156	Sherman, Colo	397	do	do	[1]15	[1]9	[1]21	......
157	Taunton, Bristol County Mass.	445	do	do	[1]15	[1]3	[1]55	......
158	Waltham, Middlesex County, Mass.	4	do	do	[1]50	[1]35	[1]10	......
159	Wilson, N. C	432	Granitite	do	[1]49.3	[1]10	[1]15	......
160	Do	433	Granite	do	[1]50	[1]32	[1]5	......
161	Winchester, Middlesex County, Mass.	57	do	do	[1]60	......	[1]20	[1]10
162	Do	58	do	do	[1]30	[1]40	[1]7	......
163	Worcester, Worcester County, Mass.	266	do	do	[1]30.8	[1]10	[1]37	......
164	Cumberland, R. I	14	Peridotite	Trap	[1]*50	([1]*)	......	......
165	Wanaque, N. J	478	Peridotite (Lherzolite)	do	......	......	......	......
166	Basic City Va	493	Quartzite	Quartzite	[1]*4.8	([1]*)	[1]94	......
167	East Providence, R. I	43	do	do	[1]7	[1]3	[1]41	......
168	Do	48	do	do	[1]5	[1]2	[1]47	......
169	Great Barrington, Berkshire County, Mass.	80	do	do	[1]7	......	[1]76.4	......
170	Lee, Berkshire County, Mass.	121	do	do	......	......	[1]98.1	......
171	Merrimac, Essex County, Mass.	329	do	do	......	......	[1]71.5	......

[a] See pages 70 and 71, Part II, for the mineral indicated by asterisk or dagger.

road-making rocks (see Part II for completed analysis)—Continued.

Augite.	Magnetite.	Apatite.	Calcite.	Olivine.	Biotite.	Chlorite.	Serpentine.	Epidote.	Pyrite.	Muscovite.	Microcline.	Garnet.	Titanite.	
	^{1}5	^{1}0.1	^{2}3			^{2}2								119
		^{1}0.1			^{1}3.9			^{2}0.2		^{1}1.3	^{1}9			120
	^{1}2	^{1}1							^{1}3	^{1}5		^{1}3		121
						^{2}10								122
	^{1}2				^{1}11			^{2}5				^{1}2		123
	^{1}1.2	^{1}.7			^{1}.1									124
					^{1}10			^{2}4			^{1}25		^{1}1	125
						^{2}12		^{2}7	^{2}1					126
					^{1}8.7			^{2}14	^{1}.1	^{1}5.4		^{1}.1	^{1}.1	127
	^{1}4	^{1}*1 *a*									^{1}20			128
	^{1}2	^{1}1			^{1}4			^{2}.1	^{1}1	^{1}6	^{1}34.5	^{1}1.3		129
		^{1}*5 *a*												130
					^{1}10					^{1}†5	(^{1}*)	(^{1}†) *a*		131
		^{1}.1			^{1}8					^{1}2				132
					^{1}5					^{1}13				133
		^{1}*2 *a*			^{1}10									134
^{1}20	^{1}*5				^{1}10				(^{1}*)				^{1}5	135
	^{1}*5	(^{1}*)			^{1}5									136
	^{1}1		^{2}2		^{1}7			^{2}2		^{1}7	^{1}6	^{1}1		137
	^{1}1				^{1}6	^{2}*2		(^{2}*)		^{1}10				138
	^{1}.1	^{1}.1			^{1}17									139
	^{1}.1	^{1}1								^{1}7	^{1}5			140
^{1}5	^{1}4	^{1}.5									^{1}60			141
	^{1}.1	^{1}.1			^{1}5					^{1}1	^{1}3			142
	^{1}.1				^{1}2	^{2}1.4		^{2}2		^{2}1.5	^{1}15			143
	^{1}.4	^{1}.1			^{1}10					^{1}3				144
	^{1}.1				^{1}8	^{2}.1								145
					^{1}7	^{2}.1		^{2}3		^{1}5	^{1}15			146
	^{1}2	^{1}.1			^{1}1					^{1}2	^{1}15		^{1}.2	147
	^{1}.5				^{1}.5						^{1}2			148
^{1}1.7		^{1}.1	^{2}4		^{1}3	^{2}6		^{2}2.4	^{2}.1				^{1}.5	149
^{1}.5	^{1}.1	^{1}.2			^{1}10	^{2}.1								150
	^{1}1	^{1}.4			^{1}7					^{1}7				151
		(^{1}*)												152
														153
			^{2}3			^{2}10							^{1}2	154
														155
	^{1}.2	^{1}.1	^{2}1		^{1}1	^{2}8				^{1}2.2	^{1}41.5			156
						^{2}1		^{2}5		^{2}20				157
						^{2}5								158
	^{1}.5				^{1}6	^{2}.2					^{1}4			159
	^{1}.3		^{2}2			^{2}1				^{1}.7				160
	^{1}*6							^{2}4					(^{1}*)	161
	^{1}2				^{1}10						^{1}10		^{1}*1 *a*	162
	^{1}1				^{1}3					^{1}5	^{1}4	^{1}2	^{2}0.2	163
	$^{1\text{-}2}$15					^{2}35								164
^{1}32	^{1}10			^{1}10			^{2}8							165
														166
	^{1}1				^{1}2	^{2}7		^{2}8		^{1}30				167
	^{1}1				^{1}1	^{1}6		^{2}6		^{1}32				168
		^{1}.2			^{1}4			^{2}2		^{1}10	^{1}0.3			169
	^{1}1	^{1}.2												170
^{1}.3			^{2}9		^{1}3	^{2}4				^{1}12				171

a See pages 70 and 71, Part II, for the mineral indicated by asterisk or dagger.

TABLE B (PART I).—*Percentage of common mineral constituents in*

	Locality.	Serial No.	Scientific name.	Common name.	Orthoclase.	Plagioclase.	Quartz.	Hornblende.
172	Methuen, Essex County, Mass.	181	Quartzite	Quartzite			[1]81	
173	Do	226	do	do			[1]66.4	[1]14
174	Monticello, Va	499	do	do	[1]35		[1]57	
175	Newport, R. I	189	do	do			[1]98.1	
176	Rowena, S. Dak	449	do	do	[1]2		[1]94	
177	Seattle, Wash	476	do	do	[1]10		[1]61	
178	Berlin, Wis	667	Rhyolite	Porphyry	[1*]88.3	[1]2	(1*)	
179	Do	668	do	do	[1*]88		(1*)	
180	Brookline, Norfolk County, Mass.	368	do	do	[1]10		[1-2]25	[2]5
181	Capital City, Colo	399	Rhyolite (breccia)	do	[1*]77.4	[1]1	(1*)	
182	Hinsdale, Colo	398	Rhyolite	do	[1*]70.9		(1*)	
183	Lake City, Colo	396	Rhyolite (breccia)	do	[1]70		[1-2]20.5	
184	Lake Shore, Colo	406	Rhyolite (tuff)	do	[1]15		[1]36.7	
185	Madison, Wis	746	Rhyolite	do	[1]56		[1]38.5	[1]5.5
186	Utley, Wis	695	do	do	[1*]91		(1*)	
187	Do	696	do	do	[1*]93		(1*)	
188	Cumberland, Md	427	Sandstone	Sandstone			[1]86.8	
189	Wilkesbarre, Pa	697	do	do			[1]86	
190	Deerpark, N. Y	344	do	do	[1*]82.9		(1*)	
191	Duanesburg, N. Y	94	do	do	[1]4	[1]1	[1]55	
192	Fort Smith, Ark	742	do	do	[1]4		[1]91	
193	Do	743	do	do	[1]4		[1]82.8	
194	Kentawa, Ky	457	do	do			[1]95.5	
195	Milton, Norfolk County, Mass.	196	do	do	[1]7	[1]1	[1]83	
196	Stonington, Miss	453	do	do	[1]5		[1]93	
197	Vicksburg, Miss	763	do	do	[1]5	[1].5	[1]22	
198	Albemarle County, Va	612	Chlorite schist	Schist			[1]31.7	
199	Alexandria County, Va	412	Mica schist	do	[1]8	[1]1	[1]63.4	
200	Asheville, N. C	469	Hornblende schist	do				[1]95.9
201	Do	705	do	do			[1]8	[1]79.5
202	Athol, Mass	88	Schist	do			[1]80	
203	Buckland, Franklin County, Mass.	52	Augite mica schist	do	[1*]74.8	[1]1	(1*)	
204	Charlottesville, Va	491	Chlorite schist	do			[1]17	
205	Do	496	Hornblende schist	do		[1]14	[1]10	[1]65
206	Do	497	do	do		[1]25		[1]58
207	Do	498	Biotite schist	do	[1]15	[1]2	[1]38	
208	Do	552	Hornblende schist	do		[1]6	[1]30	[1]46
209	Chester, Hampden County, Mass.	44	do	do			[1]21	[1]76.8
210	Clinton, Worcester County, Mass.	244	Mica schist	do	[1]9		[1]21	
211	Goat Island, Wash	508	Chlorite schist	do	[1*]5	(1*)	[1]25	
212	Great Barrington, Berkshire County, Mass.	98	Mica schist	do			[1]65	
213	Hanover, N. H	371	Hornblende schist	do		[1]15	[1]13.4	[1]60
214	Lebanon, N. H	372	do	do		[1]12	[1]10	[1]73
215	Do	373	Biotite schist	do	[1]6	[1]4	[1]51.8	
216	Lenox, Berkshire County, Mass.	10	Muscovite schist	do			[1]25	
217	Pittsfield, Berkshire County, Mass.	69	Mica schist	do			[1]30	
218	Quincy, Norfolk County, Mass.	718	Chlorite schist	do			[1-2]32.7	
219	Rockport, Me	123	Biotite schist	do			[1]38	
220	Shadwell, Va	500	Chlorite schist	do		[1]24	[1]1	
221	Webster, Worcester County, Mass.	411	Muscovite schist	do			[1]20	
222	Brookline, Norfolk County, Mass.	367	Slate	Slate			[1]9	
223	Hudson, N. Y	385	do	do			[1]72	
224	Concord, N. C	513	Syenite	Trap	[1]85.4			[1]0.4
225	Gloucester, Essex County, Mass.	30	Augite syenite	do	[1]81.5		[1]4	[1]5
226	Milton, Norfolk County, Mass.	218	do	do	[1*]62.7	(1*)	[1]13	
227	Salem, Essex County, Mass.	115	do	do	[1]58.5	[1]2.5		
228	Waltham, Middlesex County, Mass.	639	Syenite	do	[1]50.4	[1]18	[1]7	
229	California	79	Trachyte	do	[1*]95	(1*)	[1]5	
230	Saugus, Essex County, Mass.	90	do	do	[1*]80	(1*)		

road-making rocks (see Part II for completed analysis)—Continued.

Augite.	Magnetite.	Apatite.	Calcite.	Olivine.	Biotite.	Chlorite.	Serpentine.	Epidote.	Pyrite.	Muscovite.	Microcline.	Garnet.	Titanite.	
......	......	......	^{2}10	......	......	^{2}6	......	......	......	......	......	......	......	172
......	^{1}.2	......	^{2}1	......	^{1}16	......	......	^{2}2.4	......	......	......	......	......	173
......	......	......	^{2}3.5	......	......	......	......	......	......	......	......	......	......	174
......	^{1}1	^{1}.2	......	......	......	......	......	......	......	^{1}.1	......	......	......	175
......	^{1}.1	......	......	......	......	......	......	^{2}1.1	......	......	^{1}.1	......	......	176
......	......	......	^{2}9.8	......	^{1}2	^{2}.5	......	......	......	......	^{1}3	......	......	177
......	......	......	^{2}.1	......	^{1}5.1	......	......	......	......	^{2}3.2	......	......	......	178
......	^{1}1	......	......	......	^{1}9	......	......	......	......	......	......	......	......	179
......	^{1}2	......	^{2}2	......	......	^{2}16	......	^{2}40	......	......	......	......	......	180
......	^{1}1	......	^{2}6	......	......	......	......	......	......	^{1}7.6	^{1}2	......	......	181
......	^{1}2.4	^{1}.1	^{2}3	......	......	^{2}3	......	......	......	^{1}15.6	......	......	......	182
......	^{1}.5	......	^{2}5	......	^{1}1	......	......	......	......	......	......	......	......	183
......	^{1}6.5	......	^{2}12.4	......	......	......	......	......	^{2}3.4	......	......	......	......	184
......	......	......	......	......	......	......	......	......	......	......	......	......	......	185
......	^{1}2.9	......	^{2}1	......	$^{1-2}$5	......	......	......	......	......	......	......	......	186
......	^{1}2	......	^{2}1	......	$^{1\ 2}$3.8	......	......	......	......	......	......	......	......	187
......	......	......	......	......	......	......	......	......	......	^{1}2	......	......	......	188
......	^{1}2	^{1}.1	^{2}5	......	......	^{2}4	......	......	^{1}.1	^{2}1.4	......	......	......	189
......	^{1}.4	......	......	......	^{1}3.5	^{2}1.4	......	^{2}.4	......	^{1}7	......	......	......	190
......	^{1}1	......	^{2}4	......	......	^{2}3	......	......	......	......	......	......	......	191
......	^{1}.1	......	......	......	......	......	......	......	......	^{1}.3	......	......	......	192
......	^{1}.1	......	......	......	......	......	......	......	......	^{1}2	......	......	......	193
......	......	......	......	......	......	......	......	......	......	......	......	......	......	194
......	......	......	^{2}1	......	......	^{2}1	......	......	......	^{1}1	......	......	......	195
......	^{1}.4	......	......	......	......	......	......	......	......	^{1}.1	......	......	......	196
......	^{1}.2	......	......	......	......	......	......	......	......	......	......	......	......	197
......	......	......	^{2}3	......	......	^{2}22	......	^{2}42	......	......	......	......	^{1}3	198
......	......	^{1}.1	......	......	^{1}7	^{2}1	......	......	^{2}1	^{1}18	......	^{1}.4	......	199
......	^{1}1	......	......	......	......	^{2}.1	......	......	^{1}3	......	......	......	......	200
......	^{1}.4	......	......	......	......	......	......	......	......	......	......	^{1}10	^{2}2	201
......	......	......	......	......	^{1}15	......	......	......	......	......	......	......	......	202
^{1}8	......	^{1}.1	......	......	^{1}12	......	......	......	......	^{1}3	^{1}1	^{1}.1	......	203
......	......	......	......	......	^{1}.5	^{2}47	......	^{2}34.5	......	......	......	......	......	204
......	^{1}3	......	......	......	......	......	......	^{2}8	......	......	......	......	......	205
......	^{1}2	......	......	......	......	......	......	^{2}12	......	......	......	......	^{2}3	206
......	^{1}4	^{1}1	......	......	^{1}8.9	......	......	^{2}10	......	^{1}21	......	......	......	207
......	^{1}4.5	......	......	......	......	^{2}1	......	^{2}12	......	......	......	......	......	208
......	^{1}2	......	......	......	......	......	......	^{2}.1	......	......	......	......	......	209
......	......	......	......	......	^{1}12	......	......	^{2}29.9	......	^{1}28	......	......	......	210
......	......	......	^{2}2	......	......	^{2}30	......	......	^{2}.2	^{1}36.8	......	......	......	211
......	......	^{1}3	......	......	^{1}20	......	......	^{2}5	......	^{1}7	......	......	......	212
......	^{1}3	......	^{2}3.6	......	......	......	......	^{2}5	......	......	......	......	......	213
......	^{1}5	......	......	......	......	......	......	......	......	......	......	......	......	214
......	^{1}2	......	......	......	^{1}32	^{2}.2	......	......	......	......	......	^{1}4	......	215
......	^{1}5	......	......	......	^{1}3	^{2*}5	......	(^{2*})	......	^{1}60	......	^{1}2	......	216
......	^{1*}5	......	^{2}20	......	^{1}20	......	......	......	(^{1*})	^{1}25	......	......	......	217
......	^{1}6	......	^{2}6	......	^{1}.1	^{2}40	......	^{2}15	^{1}.1	......	......	......	^{1}.1	218
......	^{1}2	......	......	......	^{1}53	......	......	^{2}5	^{1}1	......	......	^{1}1	......	219
......	^{1}8	......	......	......	......	^{2}45	......	^{2}27	......	......	......	......	......	220
......	......	......	......	......	^{1}14	^{2}7	......	......	^{2}.2	^{1}58.6	......	......	......	221
......	^{1}4.5	......	^{2}8	......	......	......	......	^{2}20.2	......	^{1}63.3	......	......	......	222
......	^{1}.9	......	^{2}3	......	......	......	......	......	^{1}.1	^{1}24	......	......	......	223
^{1}3	^{1}2.5	^{1}.1	......	......	^{1}1	......	......	......	......	^{1}.1	^{1}2.5	......	......	224
^{1}4	^{1}1	......	......	......	^{1}3	......	......	......	^{1}.5	......	......	......	......	225
^{1}20	^{1}2	......	^{2}.1	......	^{1}1	......	......	......	^{2}.1	......	......	......	^{1}.1	226
^{1}22.8	^{1}5	^{1}.1	^{2}5	......	......	^{2}3	......	^{2}2	^{1}.1	......	......	......	......	227
......	......	......	^{2}5	......	......	^{2}4	......	^{2}12	......	^{1}25	......	......	......	228
......	......	......	......	......	......	......	......	......	......	......	......	......	......	229
......	$^{1\ 2}$5	......	^{2}5	......	......	^{2}10	......	......	......	......	......	......	......	230

TABLE B (PART II).—*Percentage of rarer*

[The small numerals placed above the percentage figures denote, respectively, whether the mineral posed, and the secondary those resulting from subsequent change. In some cases the separate deter- two is given in one column and an asterisk or dagger indicates the other mineral.]

	Locality.	Serial No.	Scientific name.	Common name.	Fluorite.	Microperthite.	Hematite.	Zircon.
1	Charlottesville, Va	364	Amphibolite	Trap				
2	Do	548	do	do				
3	Do	549	do	do				
4	Do	550	do	do				
5	Do	551	do	do				
6	Wallingford, Vt	726	do	do				
7	Cienfuegos, Cuba	471	Andesite	do				
8	Lake City, Colo	407	do	do				
9	Do	408	do	do				
10	Ansonia, Conn	354	Basalt	do				
11	Boundbrook, N. J	357	do	do				
12	Do	252	do	do				
13	Byram Station, N. J	251	do	do				
14	Deerfield, Franklin County, Mass.	268	do	do				
15	Guttenberg, N. J	73	do	do				
16	Morganton, N. C	535	do	do				
17	Seattle, Wash	477	do	do				
18	Great Notch, N. J	260	do	do				
19	Somerville, N. J	637	Basalt tuff	do			^{2}5.0	
20	Do	669	do	do				
21	Wilmington, N. J	250	Basalt	do				
22	Salisbury, Essex County, Mass.	33	Camptonite	do				
23	Birmingham, Ala	391	Chert	Chert				
24	Do	392	do	do				
25	Do	393	do	do				
26	Bluesprings, Nebr	360	do	do				
27	Elco, Ill	437	do	do				
28	Do	584	do	do				
29	Graytown, Tenn	338	do	do				
30	Hot Springs, Ark	438	do	do				
31	San Jose, Cal	538	do	do			^{2}2.5	
32	Amherst, Hampshire County, Mass.	78	Diabase	Trap				
33	Do	82	do	do				
34	Brookline, Norfolk County, Mass.	7	do	do				
35	Do	369	Diabase (altered)	do			^{2}3.2	
36	Do	23	Diabase	do				
37	Charlottesville, Va	494	Diabase (uralitic)	do				
38	Coweta County, Ga	415	Diabase	do				
39	Deerfield, Franklin County, Mass.	76	do	do				
40	Dickerson, Md	363	do	do				
41	Everett, Middlesex County, Mass.	2	do	do				
42	Great Notch, N. J	261	do	do				
43	Haverstraw, N. Y	49	do	do				
44	Holyoke, Hampden County, Mass.	66	do	do				
45	Hydepark, Norfolk County, Mass.	9	do	do				
46	Ipswich, Essex County, Mass.	61	Diabase (uralitic)	do				
47	Lynn, Essex County, Mass.	21	Diabase	do				
48	Do	29	do	do				
49	Malden, Middlesex County, Mass.	270	do	do				
50	Medford, Middlesex County, Mass.	20	do	do				
51	Meriden, Conn	11	do	do				
52	Do	71	do	do				
53	Do	467	do	do				
54	Middle Valley, N. J	405	do	do				
55	Milton, Norfolk County, Mass.	219	Diabase (uralitic)	do				
56	Monson, Hampden County, Mass.	68	Diabase	do				
57	Newbury, Essex County, Mass.	31	do	do				
58	New Providence, N. J	350	do	do				
59	Newton, Middlesex County, Mass.	6	do	do				

mineral constituents in road-making rocks.

is primary or secondary, the primary minerals being the original minerals of which the rock is com-
mination of two minerals in the same rock is impracticable, therefore the total percentage of the

Allanite.	Rutile.	Limonite.	Glaucophane.	Rock glass.	Tourmaline.	Kaolin.	Scolecite.	Natrolite.	Hypersthene.	Nephelite.	Opal.	
												1
		[2]0.1										2
												3
		[2]0.1										4
												5
												6
				[1]15.0								7
		[2]1.5										8
												9
												10
				[1]10								11
				[1]7		[2]19						12
												13
				[1]12		[2]15						14
												15
												16
												17
				[1]24								18
				[1]72								19
		[2]3.7		[1]80								20
				[1]20		[2]15						21
												22
		[2]0.5										23
		[2]0.5										24
		[2]0.5										25
		[2]3.8										26
		[2]2										27
		[2]3.3										28
		[2]3										29
					[1]0.1							30
						[2]6.8						31
												32
												33
												34
												35
												36
												37
												38
												39
												40
												41
				[1]22								42
												43
												44
												45
												46
												47
												48
				[1]28								49
												50
												51
		[2]2				[2]10						52
		[2]1					[2]5					53
						[2]20						54
												55
												56
												57
		[2]1				[2]40						58
												59

TABLE B (PART II).—*Percentage of rarer mineral*

	Locality.	Serial No.	Scientific name.	Common name.	Fluorite.	Microperthite.	Hematite.	Zircon.
60	Quincy, Norfolk County, Mass.	18	Diabase	Trap				
61	Do	299	Diabase (uralitic)	do				
62	Roberts Station, Ga	420	Diabase	do				
63	Rockhill, Pa	731	do	do				
64	Rockland Lake, N. Y	335	do	do				
65	Saugus, Essex County, Mass.	22	do	do				
66	Do	25	do	do				
67	Do	28	do	do				
68	Do	32	do	do				
69	Do	84	do	do				
70	Shadyside, N. J	566	do	do				
71	Somerville, Middlesex County, Mass.	19	Diabase (gabbroitic)	do				
72	Do	75	Diabase	do				
73	Sterling, Worcester County, Mass.	83	do	do				
74	Ware, Hampshire County, Mass.	64	do	do				
75	Westfield, Hampden County Mass.	517	do	do				
76	West Springfield, Hampden County, Mass.	12	do	do				
77	Do	67	do	do				
78	Do	91	do	do				
79	Do	93	do	do				
80	Winchester, Middlesex County, Mass.	59	do	do				
81	Asheville, N. C	628	Diorite	do				[1]1
82	Beverly, Essex County, Mass.	117	do	do				
83	Campo, Fla	472	do	do				
84	Charlottesville, Va	490	do	do				
85	Delaware River, Pa	352	do	do				
86	Havana, Cuba	575	do	do				
87	Lake City, Colo	409	do	do				[1]0.1
88	Millville, Worcester County, Mass.	227	do	do				
89	Newbury, Essex County, Mass.	35	do	do				
90	Providence, R. I	659	Augite-diorite	do				
91	Salem, Essex County, Mass.	1	do	do				
92	Do	611	Diorite (vogesite)	do				
93	Watertown, Middlesex County, Mass.	96	Diorite	do				
94	Winchester, Middlesex County, Mass.	56	do	do				
95	Do	304	do	do				
96	Canton, Ga	583	Eclogite	Garnet rock				
97	Glen Mills, Pa	254	do	do				
98	Ashboro, N. C	514	Felsite	Porphyry				
99	Boston, Suffolk County, Mass.	40	do	do				
100	Lynn, Essex County, Mass.	24	do	do				
101	Do	27	do	do			[1]10	
102	Quincy, Norfolk County, Mass.	72	do	do				
103	Revere, Suffolk County, Mass.	3	do	do				
104	Do	124	do	do				
105	Rowley, Essex County, Mass.	97	do	do				
106	Saugus, Essex County, Mass.	86	do	do				
107	Do	87	do	do				
108	Weymouth, Norfolk County, Mass.	92	do	do				
109	Bergen, N. J	249	Gabbro	Trap				
110	Cortland, N. Y	195	do	do				
111	Lambertsville, N. J	253	Uralite gabbro	do				
112	Do	387	Gabbro	do				
113	Rocky Hill, N. J	248	do	do				
114	Do	285	Uralite gabbro	do				
115	Athol, Worcester County, Mass.	88	Gneiss	Gneiss				[1]0.1
116	Albemarle County, Va	613	do	do				[1]0.1
117	Atlanta, Ga	422	Hornblende-gneiss	do				
118	Auburn, Worcester County, Mass.	203	Gneiss	do				

constituents in road-making rocks—Continued.

Allanite.	Rutile.	Limonite.	Glaucophane.	Rock glass.	Tourmaline.	Kaolin.	Scolecite.	Natrolite.	Hypersthene.	Nephelite.	Opal.	
......	......	......	......	......	......	......	......	......	......	......	......	60
......	......	......	......	......	......	......	......	......	......	......	......	61
......	......	[2]1.4	......	......	......	[2]3	......	......	......	......	......	62
......	......	......	......	......	......	......	......	......	......	......	......	63
......	......	......	......	......	......	[2]7	......	......	......	......	......	64
......	......	......	......	......	......	......	......	......	......	......	......	65
......	......	......	......	......	......	......	......	......	......	......	......	66
......	......	......	......	......	......	......	......	......	......	......	......	67
......	......	......	......	......	......	......	......	......	......	......	......	68
......	......	......	......	......	......	......	......	......	......	......	......	69
......	......	......	......	......	......	......	[2]7	......	......	......	......	70
......	......	......	......	......	......	[2]14	......	......	......	......	......	71
[1]5	......	......	......	......	......	......	......	......	......	......	......	72
......	......	......	......	......	......	[2]0.1	......	......	......	......	......	73
......	......	......	......	......	......	......	......	......	......	......	......	74
......	......	......	......	......	......	[2]3	......	......	......	......	......	75
......	......	......	......	......	......	......	......	......	......	......	......	76
......	......	......	......	......	......	......	......	......	......	......	......	77
......	......	......	......	......	......	......	......	......	......	......	......	78
......	......	......	......	......	......	......	......	......	......	......	......	79
......	......	......	......	......	......	......	......	......	......	......	......	80
......	......	[2]0.1	......	......	......	......	......	......	......	......	......	81
......	......	......	......	......	......	[2]16	......	......	......	......	......	82
......	......	......	......	......	......	[2]2	......	......	......	......	......	83
......	......	......	......	......	......	......	......	......	......	......	......	84
......	......	[2]0.2	......	......	......	[2]10	......	......	......	......	......	85
......	......	......	......	......	......	[2]*57.8	......	([2]*)	......	......	......	86
......	......	......	......	......	......	......	......	......	......	......	......	87
......	......	......	......	......	......	[2]1.6	......	......	......	......	......	88
......	......	......	......	......	......	......	......	......	......	......	......	89
......	......	......	......	......	......	[2]11	......	......	......	......	......	90
......	......	......	......	......	......	......	......	......	......	......	......	91
......	......	......	......	......	......	[2]1	......	......	......	......	......	92
......	......	......	......	......	......	......	......	......	......	......	......	93
......	......	......	......	......	......	......	......	......	......	......	......	94
......	......	......	......	......	......	[2]15	......	......	......	......	......	95
......	......	......	......	......	......	......	......	......	......	......	......	96
......	......	......	......	......	......	......	......	......	......	......	......	97
......	......	......	......	......	......	......	......	......	......	......	......	98
......	......	......	......	......	......	......	......	......	......	......	......	99
......	......	......	......	......	......	......	......	......	......	......	......	100
......	......	......	......	......	......	......	......	......	......	......	......	101
......	......	......	......	......	......	......	......	......	......	......	......	102
......	......	......	......	......	......	......	......	......	......	......	......	103
......	......	[2]2	......	......	......	[2]0.1	......	......	......	......	......	104
......	......	......	......	......	......	......	......	......	......	......	......	105
......	......	......	......	......	......	......	......	......	......	......	......	106
......	......	......	......	......	......	......	......	......	......	......	......	107
......	......	......	......	......	......	......	......	......	......	......	......	108
......	......	[2]1	......	......	......	[2]6	......	......	......	......	......	109
......	......	......	......	......	......	......	......	......	[1]14	......	......	110
......	......	......	......	......	......	[2]30	......	......	......	......	......	111
......	......	......	......	......	......	[2]18.2	......	......	......	......	......	112
......	......	......	......	......	......	[2]3	......	......	......	......	......	113
......	......	......	......	......	......	[2]35.8	......	......	......	......	......	114
......	......	......	......	......	......	[2]0.1	......	......	......	......	......	115
......	......	[2]1	......	......	......	......	......	......	......	......	......	116
......	......	......	......	......	......	......	......	......	......	......	......	117
......	......	[2]0.1	......	......	......	[2]0.1	......	......	......	......	......	118

TABLE B (PART II).—*Percentage of rarer mineral*

	Locality.	Serial No.	Scientific name.	Common name.	Fluorite.	Microperthite.	Hematite.	Zircon.
119	Augusta, Ga	419	Granite-gneiss	Gneiss				[1]0.1
120	Ashby, Middlesex County, Mass.	118	Gneiss	do				
121	Dartmouth, Bristol County, Mass.	77	do	do		([1]*)		
122	Duxbury, Plymouth County, Mass.	5	do	do				
123	Lee, Berkshire County, Mass.	51	Hornblende gneiss	do				
124	Little Falls, N. Y	532	Granite gneiss	do				
125	Marion, Plymouth County, Mass.	13	Gneiss	do				
126	Newburyport, Essex County, Mass.	37	do	do				
127	Occoquan, Va	581	do	do				
128	Round Island, N. Y	100	do	do				([1]*)
129	Toccoa, Ga	468	Granite gneiss	do				[1].1
130	Uxbridge, Worcester County, Mass.	63	Gneiss	do				([1]*)
131	Westport, Bristol County, Mass.	81	do	do			([1]†)	
132	Ashby, Middlesex County, Mass.	60	Granite	Granite				
133	Asheville, N. C	627	do	do				
134	Beverly, Essex County, Mass.	62	Hornblende granite	do				([1]*)
135	Do	74	Granite	do				
136	Do	89	Hornblende granite	do				
137	Clinton, Worcester County, Mass.	242	Gneissoid granite	do				
138	Do	279	Granite	do				[1].1
139	Columbia, S. C	374	do	do				
140	Concord, N. C	747	do	do				
141	Do	436	do	do				[1].5
142	Elberton, Ga	416	do	do				
143	Elm City, N. C	431	do	do				
144	Fitchburg, Worcester County, Mass.	256	do	do				
145	Gloucester, Essex County, Mass.	26	Hornblende granitite	do		[1]10		
146	Hanover, N. H	370	Gneissoid granite	do		[1]10		
147	Lithonia, Ga	421	Granite	do				
148	Los Angeles, Cal	578	do	do				[1].1
149	Northampton, Hampshire County, Mass.	8	Hornblende granitite	do				
150	Orange, Franklin County, Mass.	39	do	do				[1].1
151	Pacolet, S. C	375	Granite	do				
152	Quincy, Norfolk County, Mass.	17	do	do		([1]†)		
153	Rockport, Essex County, Mass.	34	do	do				[1]2
154	Saugus, Essex County, Mass.	15	do	do				
155	Do	32	do	do				
156	Sherman, Colo	397	do	do			[2]1	
157	Taunton, Bristol County, Mass.	445	do	do				
158	Waltham, Middlesex County, Mass.	4	do	do				
159	Wilson, N. C	432	Granitite	do				
160	Do	433	do	do			[2]2	
161	Winchester, Middlesex County, Mass.	57	do	do				
162	Do	58	do	do			([1]*)	
163	Worcester, Worcester County, Mass.	266	do	do	[1]0.2			
164	Cumberland, R. I	14	Peridotite	Trap				
165	Wanaque, N. J	478	Peridotite (Lherzolite)	do				
166	Basic City, Va	493	Quartzite	Quartzite				[1]0.2
167	East Providence, R. I	43	do	do				
168	Do	48	do	do				
169	Great Barrington, Berkshire County, Mass.	80	do	do				[1].1
170	Lee, Berkshire County, Mass.	121	do	do				[1].2
171	Merrimac, Essex County, Mass.	329	do	do				[1].1

constituents in road-making rocks—Continued.

Allanite.	Rutile.	Limonite.	Glaucophane.	Rock glass.	Tourmaline.	Kaolin.	Scolecite.	Natrolite.	Hypersthene.	Nephelite.	Opal.	
		[2]2										119
						[2]2.5						120
	[1]1											121
												122
												123
												124
												125
												126
		[2]0.1										127
			[1]35									128
												129
												130
												131
						[2]1						132
		[2]2										133
												134
												135
												136
												137
		[2]2										138
						[2]7						139
		[2]3.9										140
												141
[2].5						[2]1						142
		[2]1.5										143
						[2]2						144
												145
												146
												147
		[2].6				[2]2.7						148
						[2]14						149
												150
												151
												152
												153
												154
												155
												156
		[2]1										157
												158
						[2]15						159
						[2]7						160
												161
												162
						[2]7						163
												164
									[1]40			165
		[2]1										166
		[2]1										167
												168
												169
		[2].5										170
	[1]0.1											171

TABLE B (PART II).—*Percentage of rarer mineral*

	Locality.	Serial No.	Scientific name.	Common name.	Fluorite.	Microperthite.	Hematite.	Zircon.
119	Augusta, Ga	419	Granite-gneiss	Gneiss				[1]0.1
120	Ashby, Middlesex County, Mass.	118	Gneiss	do				
121	Dartmouth, Bristol County, Mass.	77	do	do		([1]*)		
122	Duxbury, Plymouth County, Mass.	5	do	do				
123	Lee, Berkshire County, Mass.	51	Hornblende gneiss	do				
124	Little Falls, N. Y	532	Granite gneiss	do				
125	Marion, Plymouth County, Mass.	13	Gneiss	do				
126	Newburyport, Essex County, Mass.	37	do	do				
127	Occoquan, Va	581	do	do				
128	Round Island, N. Y	100	do	do				([1]*)
129	Toccoa, Ga	468	Granite gneiss	do				[1].1
130	Uxbridge, Worcester County, Mass.	63	Gneiss	do				([1]*)
131	Westport, Bristol County, Mass.	81	do	do			([1]†)	
132	Ashby, Middlesex County, Mass.	60	Granite	Granite				
133	Asheville, N. C	627	do	do				
134	Beverly, Essex County, Mass.	62	Hornblende granite	do				([1]*)
135	Do	74	Granite	do				
136	Do	89	Hornblende granite	do				
137	Clinton, Worcester County, Mass.	242	Gneissoid granite	do				
138	Do	279	Granite	do				[1].1
139	Columbia, S. C	374	do	do				
140	Concord, N. C	747	do	do				
141	Do	436	do	do				[1].5
142	Elberton, Ga	416	do	do				
143	Elm City, N. C	431	do	do				
144	Fitchburg, Worcester County, Mass.	256	do	do				
145	Gloucester, Essex County, Mass.	26	Hornblende granitite	do		[1]10		
146	Hanover, N. H	370	Gneissoid granite	do		[1]10		
147	Lithonia, Ga	421	Granite	do				
148	Los Angeles, Cal	578	do	do				[1].1
149	Northampton, Hampshire County, Mass.	8	Hornblende granitite	do				
150	Orange, Franklin County, Mass.	39	do	do				[1].1
151	Pacolet, S. C	375	Granite	do				
152	Quincy, Norfolk County, Mass.	17	do	do		([1]†)		
153	Rockport, Essex County, Mass.	34	do	do				[1]2
154	Saugus, Essex County, Mass.	15	do	do				
155	Do	32	do	do				
156	Sherman, Colo	397	do	do			[2]1	
157	Taunton, Bristol County, Mass.	445	do	do				
158	Waltham, Middlesex County, Mass.	4	do	do				
159	Wilson, N. C	432	Granitite	do				
160	Do	433	do	do			[2]2	
161	Winchester, Middlesex County, Mass.	57	do	do				
162	Do	58	do	do			([1]*)	
163	Worcester, Worcester County, Mass.	266	do	do	[1]0.2			
164	Cumberland, R. I	14	Peridotite	Trap				
165	Wanaque, N. J	478	Peridotite (Lherzolite)	do				
166	Basic City, Va	493	Quartzite	Quartzite				[1]0.2
167	East Providence, R. I	43	do	do				
168	Do	48	do	do				
169	Great Barrington, Berkshire County, Mass.	80	do	do				[1].1
170	Lee, Berkshire County, Mass.	121	do	do				[1].2
171	Merrimac, Essex County, Mass.	329	do	do				[1].1

constituents in road-making rocks—Continued.

Allanite.	Rutile.	Limonite.	Glaucophane.	Rock glass.	Tourmaline.	Kaolin.	Scolecite.	Natrolite.	Hypersthene.	Nephelite.	Opal.	
		[2]2										119
						[2]2.5						120
	[1]1											121
												122
												123
												124
												125
												126
		[2]0.1										127
			[1]35									128
												129
												130
												131
						[2]1						132
		[2]2										133
												134
												135
												136
												137
		[2]2										138
						[2]7						139
		[2]3.9										140
												141
[2].5						[2]1						142
		[2]1.5										143
						[2]2						144
												145
												146
												147
		[2].6				[2]2.7						148
						[2]14						149
												150
												151
												152
												153
												154
												155
												156
		[2]1										157
												158
						[2]15						159
						[2]7						160
												161
												162
						[2]7						163
												164
									[1]40			165
		[2]1										166
		[2]1										167
												168
												169
		[2].5										170
	[1]0.1											171

TABLE B (PART II).—*Percentage of rarer mineral*

	Locality.	Serial No.	Scientific name.	Common name.	Fluorite.	Microperthite.	Hematite.	Zircon.
172	Methuen, Essex County, Mass.	181	Quartzite	Quartzite				[1].1
173	Do	226	do	do				
174	Monticello, Va	499	do	do				
175	Newport, R. I	189	do	do				[1].1
176	Rowena, S. Dak	449	do	do			[2]2.6	[1].1
177	Seattle, Wash	476	do	do				
178	Berlin, Wis	667	Rhyolite	Porphyry				
179	Do	668	do	do				
180	Brookline, Norfolk County, Mass.	368	do	do				
181	Capital City, Colo	399	Rhyolite (breccia)	do			[2]5	
182	Hinsdale, Colo	398	Rhyolite	do			[2]5	
183	Lake City, Colo	396	Rhyolite (breccia)	do				
184	Lake Shore, Colo	406	Rhyolite (tuff)	do				
185	Madison, Wis	746	Rhyolite	do				
186	Utley, Wis	695	do	do				[1].1
187	Do	696	do	do				[1].2
188	Cumberland, Md	427	Sandstone	Sandstone				
189	Wilkesbarre, Pa	697	do	do				[1].2
190	Deerpark, N. Y	344	do	do				[1].1
191	Duanesburg, N. Y	94	do	do				
192	Fort Smith, Ark	742	do	do				[1].1
193	Do	743	do	do				[1].1
194	Kentawa, Ky	457	do	do				
195	Milton, Norfolk County, Mass.	196	do	do				[1].1
196	Stonington, Miss	453	do	do				
197	Vicksburg, Miss	753	do	do				
198	Albemarle County, Va	612	Chlorite schist	Schist				
199	Alexandria County, Va	412	Mica schist	do			[2].1	
200	Asheville, N. C	469	Hornblende schist	do				
201	Do	705	do	do				
202	Athol, Mass	88	Schist	do				
203	Buckland, Franklin County, Mass.	52	Augite mica schist	do				
204	Charlottesville, Va	491	Chlorite schist	do				
205	Do	496	Hornblende schist	do				
206	Do	497	do	do				
207	Do	498	Biotite schist	do				[1].1
208	Do	552	Hornblende schist	do				
209	Chester, Hampden County, Mass.	44	do	do				
210	Clinton, Worcester County, Mass.	244	Mica schist	do				.1
211	Goat Island, Wash	508	Chlorite schist	do				
212	Great Barrington, Berkshire County, Mass.	98	Mica schist	do				
213	Hanover, N. H	371	Hornblende schist	do				
214	Lebanon, N. H	372	do	do				
215	Do	373	Biotite schist	do				
216	Lenox, Berkshire County, Mass.	10	Muscovite schist	do				
217	Pittsfield, Berkshire County, Mass.	69	Mica schist	do				
218	Quincy, Norfolk County, Mass.	718	Chlorite schist	do				
219	Rockport, Me	123	Biotite schist	do				
220	Shadwell, Va	500	Chlorite schist	do				
221	Webster, Worcester County, Mass.	411	Muscovite schist	do				
222	Brookline, Norfolk County, Mass.	367	Slate	Slate				
223	Hudson, N. Y	385	do	do				
224	Concord, N. C	513	Syenite	Trap				
225	Gloucester, Essex County, Mass.	30	Augite syenite	do				
226	Milton, Norfolk County, Mass.	218	do	do				
227	Salem, Essex County, Mass.	115	do	do				
228	Waltham, Middlesex County, Mass.	639	Syenite	do				[1].1
229	California	79	Trachyte	do				
230	Saugus, Essex County, Mass.	90	do	do				

constituents in road-making rocks—Continued.

Allanite.	Rutile.	Limonite.	Glaucophane.	Rock glass.	Tourmaline.	Kaolin.	Scolecite.	Natrolite.	Hypersthene.	Nephelite.	Opal.	
		[2]2.9										172
												173
		[2]4.5										174
		[2].5										175
												176
		[2]8.7				[2]5						177
		[2]1.3										178
		[2]2										179
												180
												181
												182
		[2]3										183
		[2]1				[2]25						184
												185
												186
												187
		[2]11.1			[1]0.1							188
		[2]1.2										189
		[2]4.3										190
		[2]12				[2]20						191
	[1].1	[2].4				[2]4						192
		[2]7				[2]4						193
		[2]4.5										194
		[2]2				[2]3.9						195
		[2]1				[2].5						196
		[2]1									[2]71.3	197
		[2]1										198
												199
												200
		[2].1										201
		[2]5										202
												203
		[2]1.										204
												205
												206
												207
		[2].5										208
		[2].1										209
												210
		[2]1										211
												212
												213
												214
												215
												216
												217
												218
												219
												220
		[2].2										221
												222
												223
						[2]5						224
										[1]1		225
						[2]1						226
						[2]1						227
		[2]1										228
												229
												230

FORMS FOR REPORTING RESULTS OF TESTS, ETC.

INSTRUCTIONS FOR SELECTING AND SHIPPING SAMPLES.

In order to have road materials tested in the laboratory of the Department of Agriculture, the instructions below must be carefully followed:

1. All samples should be selected to represent as nearly as possible an average of the material.
2. A sample of rock for laboratory test must consist of stones which will pass through a 3-inch but not through an inch and a half ring—excepting one piece, which should measure, approximately, 4 by 6 inches on one face and be about 3 inches thick. The whole sample should weigh not less than 30 pounds. It is desired that samples of rock be shipped in burlap bags.
3. A sample of gravel must weigh not less than 25 pounds, and should not contain stones over 1 inch in diameter. Such samples must be shipped in boxes, sufficiently tight to prevent the finer material from sifting out.
4. A sample of paving brick must contain 36 whole bricks, or 24 blocks, which must be securely packed in a box for shipment.
5. A blank form and addressed tag envelope will be supplied by the Department for each sample. The blank form must be filled and placed in the tag envelope, which must be used as the address for the sample. It is essential that these blank forms be filled with the utmost care, as they are filed as records of the samples.
6. The Agricultural Department desires to keep a record of the actual wear on roads built of the materials tested. If the material which this sample represents has been, or is about to be, used on roads, this laboratory desires to be informed of the addresses of those in charge of the construction and maintenance of such roads.
7. Samples must be shipped, freight or expressage prepaid, and bills of lading or express receipts forwarded by mail to the Road Material Laboratory, Bureau of Chemistry, U. S. Department of Agriculture, Washington, D. C.

SHIPPING BLANK.

1. The sample about to be shipped is from the State of ———. County ———. Town or city ———.
2. It comes from the property of ———.
3. If rock, about how many cubic yards are available? ———.
4. Name and address of person sending sample: ———.
5. Date of shipment: ———, 19—.
6. Is test desired by a private person, municipality, or company? ———.
7. Name of person, municipality, or company: ———.
8. Is material intended for general sale to the public? ———.
9. Has material been used on roads? If so, where? ———.

If a judgment of sample's fitness for a particular road is desired, further details must be given, and Form No. 3 will be mailed to you for this purpose.

DO NOT WRITE BELOW THIS LINE.

Date received at laboratory: ———, 19—. Sample No. ——.

Tests made on sample: ———. Abrasion No. ——. Result: ———. Cementation No. ——. Result: ———. Toughness No. ——. Result: ———. Hardness No. ——. Result: ———. Density No. ——. Result: ———.

Name of material: ———.

Other tests: ———.

Condition of sample: ———.

REMARKS: ———.

REPORT ON PHYSICAL TESTS.

Report on sample No. —— of road material from ———.
Made at the request of ———.
Material: ———.

Results of physical tests on sample submitted, and a comparison with highest and lowest results obtained on material of a similar nature.

Determinations.	Results on sample submitted.	Results on similar materials.	
		Highest.	Lowest.
Specific gravity			
Weight per cubic foot	lbs.	lbs.	lbs.
Water absorbed per cubic foot	lbs.	lbs.	lbs.
Per cent of wear			
Hardness			
Toughness			
Cementing value			

An explanation of tests is given in the accompanying circular.

Remarks:

REPORT ON CHEMICAL AND MINERAL COMPOSITION.

Chemical and mineral composition of sample No. ——— of road material from ———.
Made at the request of ———.
Character of material: ———.

Essential minerals:

Accessory minerals:

Secondary minerals:

Name:

Chemical Analysis:
Silica (SiO_2) ....
Alumina (Al_2O_3) ....
Iron oxid (Fe_2O_3) ....
Manganese oxid (MnO_2) ....
Titanium oxid (TiO_2) ....
Lime (CaO) ....
Magnesia (MgO) ....
Alkalies $\left\{\begin{array}{l}K_2O \text{ ....}\\ Na_2O \text{ ....}\end{array}\right.$
Loss on ignition ....
Insoluble in HCl ....
Water at 105° C ....
Water above 105° C ....
....
....
....
....
Total ....

Remarks:

Date: ———.

ABRASION TEST.

Data.	Material used in test.	Sizes of material after test.				
Inches Centimeters	2.4–1.2 6–3	2.4–1.2 6–3	1.2–$\frac{1}{16}$ 3–.16	$\frac{1}{16}$–$\frac{1}{100}$ .16–.025	All under $\frac{1}{16}$.	All detritus.
Weight in ounces						
Weight in grams						
Number of pieces						
Per cent						

Serial No. ———. Name of material ———.
Locality ———.
Date of test ———. Department's coefficient of wear ———.
Made by ———. French coefficient of wear ———.
Percentage of wear ———.

REMARKS:

RATTLER TEST OF PAVING BRICK.

GENERAL REPORT.

Report of Rattler tests on sample No. ——— of paving brick from ———.
Made at request of ———.
Trade name of brick ———.
Condition of sample ———.

Determinations.	Average of charge.		Greatest individual loss.	Least individual loss.
	Test No. 1.	Test No. 2.		
Loss in per cent at ——— revolutions				
Loss in per cent from 1,200–1,800 revolutions				
Loss in per cent at 1,800 revolutions				

Average loss at 1,800 revolutions, in per cent ———.
Highest and lowest results obtained up to the present date on other paving brick ———.
Rattler loss in per cent at —— revolutions ———. Average of charge ———.
Rattler loss in per cent from 1,200–1,800 revolutions ——. " " ———.
Rattler loss in per cent at 1,800 revolutions ———. " " ———

REMARKS:

Date ———.

DETAILED REPORT.

Serial No. ———. Trade name of brick ———.
Shipped from ———.
Date of test ———. Made by ———.
Condition of sample when received ———.
Size of Rattler ———. Speed of Rattler ——— R. P. M.
Charge ———. Shot ———.
Brick ———.
Size of brick ———.

RESULTS.

Number of brick.	Original weight, kgs.	Loss in Rattler.					
		—— rev.		1,200 rev.		1,800 rev.	
		In kgs.	In per cent.	In kgs.	In per cent.	In kgs.	In per cent.
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
Average							
Greatest							
Least							

Remarks:

FOR RECORDING VOLUME AND CHARACTER OF TRAFFIC.

N. B.—This form should be used for recording the traffic of one road only.

Each variety of traffic enumerated below should be kept on separate slips of paper and the total number of each variety placed in its proper square after a record of twenty-four consecutive hours has been kept. Less than a week's record is unsatisfactory, and it is best that a record should cover as many seasons of the year as possible. Additional forms will be supplied on application.

1	Loaded one-horse wagon														
2	Unloaded one-horse wagon														
3	Loaded two-horse wagon														
4	Unloaded two-horse wagon														
5	Loaded four-horse wagon														
6	Unloaded four-horse wagon														
7	One-horse pleasure vehicle														
8	Two-horse pleasure vehicle														
9	Rubber-tired vehicle														
10	Unharnessed horse or shod ox														
11	Excessively heavy vehicle[a]														
12	Year														
13	Day														
14	Month.														

[a] 11 is intended for very heavy vehicles such as carry ore, stone, brick, timber, etc.

Traffic on road in State of ——, county ——, town ——.

Name of road, or between what two points was record taken ——.

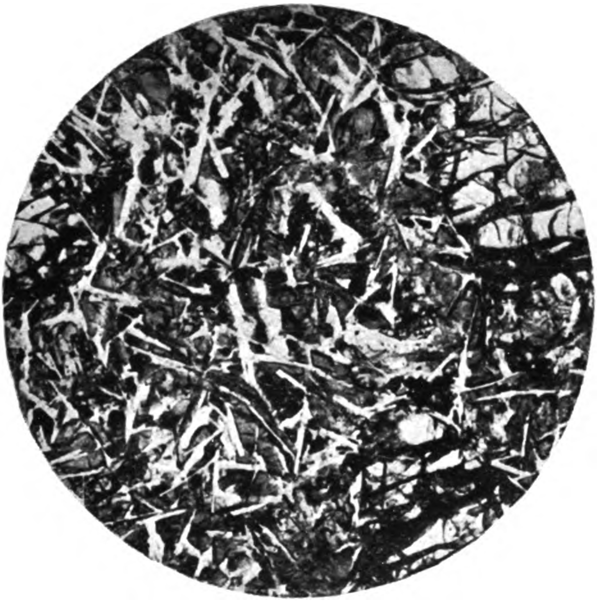

FIG. 1.—BASALT (TRAP).

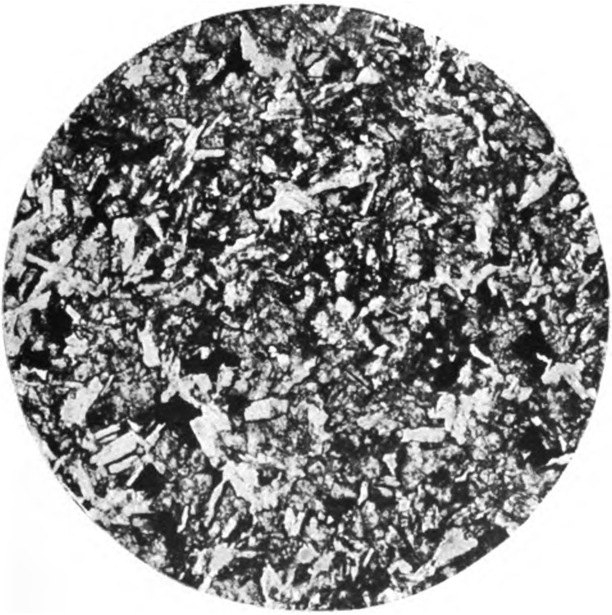

FIG. 2.—DIABASE (TRAP).

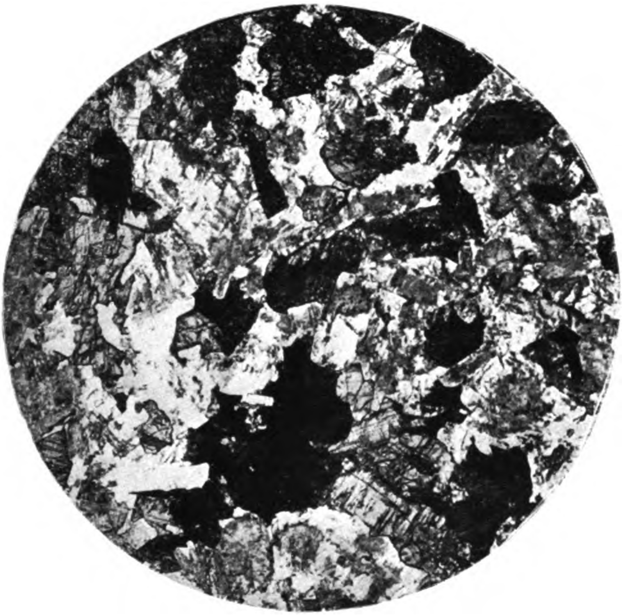

FIG. 1.—GABBRO.

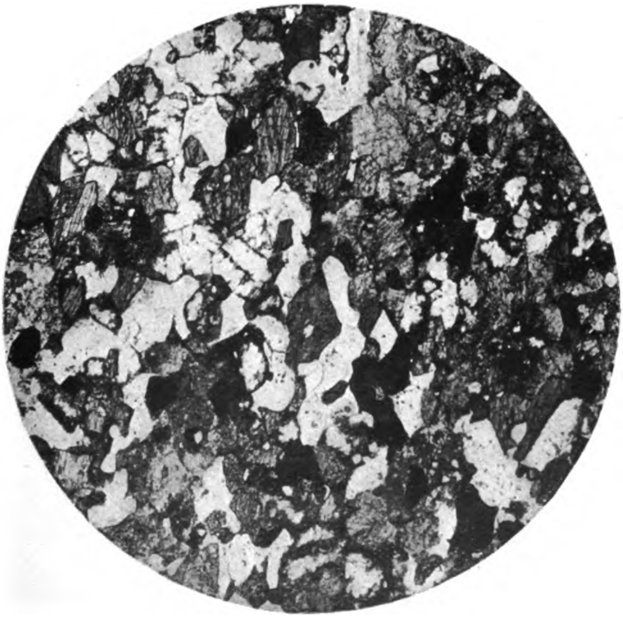

FIG. 2.—DIORITE.

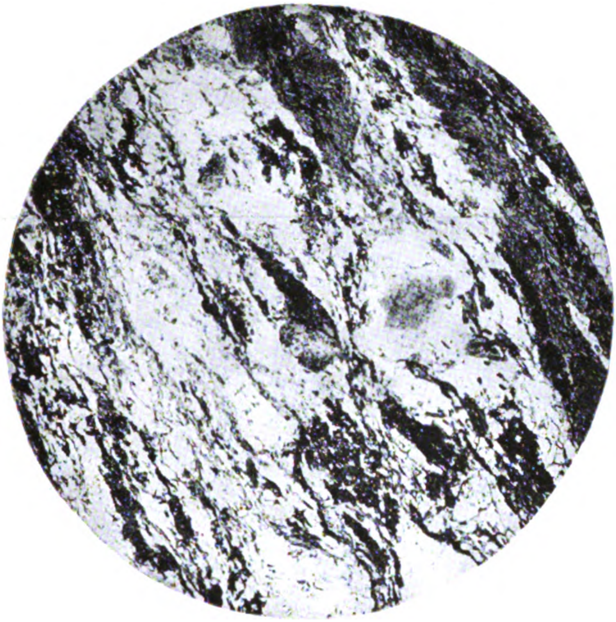

FIG. 1.—GNEISS.

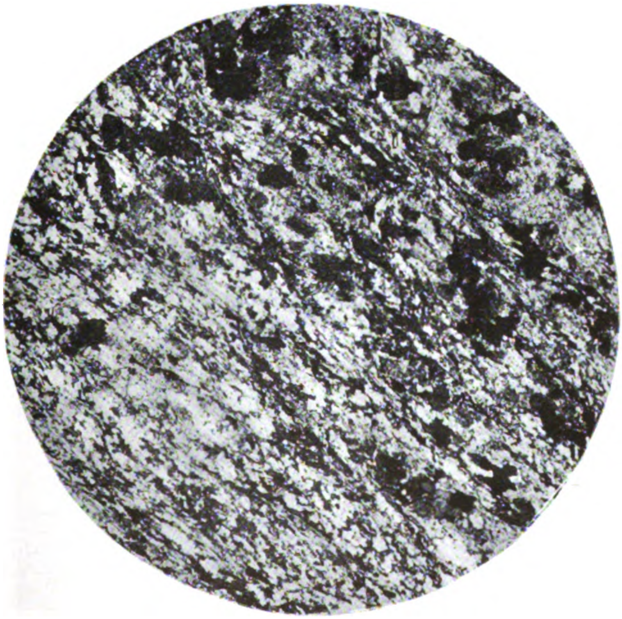

FIG. 2.—MICA-SCHIST.

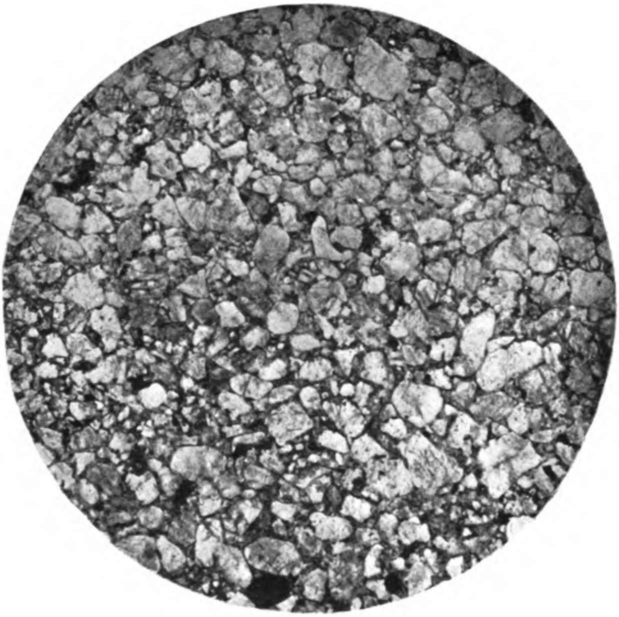

FIG. 1.—QUARTZITE.

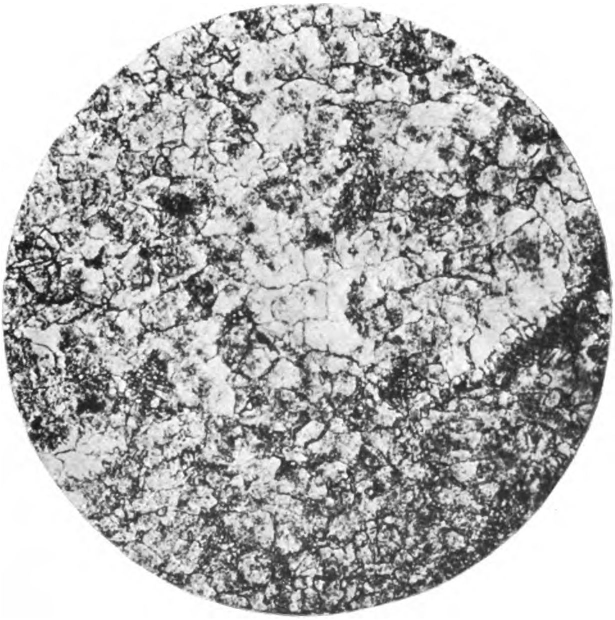

FIG. 2.—LIMESTONE.

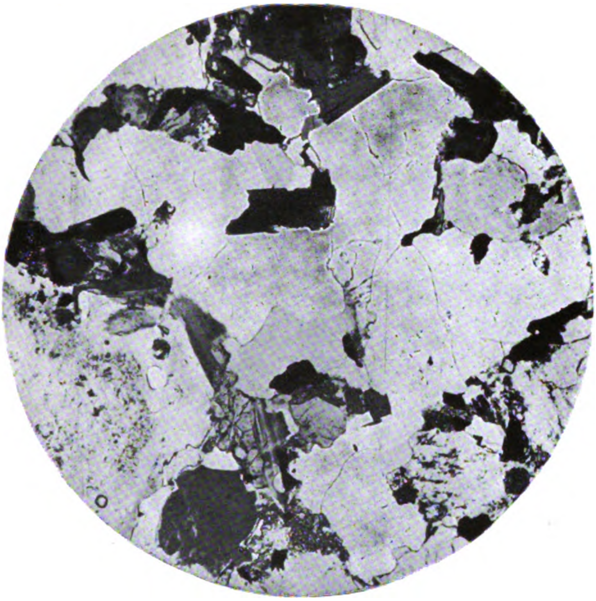

FIG. 1.—GRANITE.

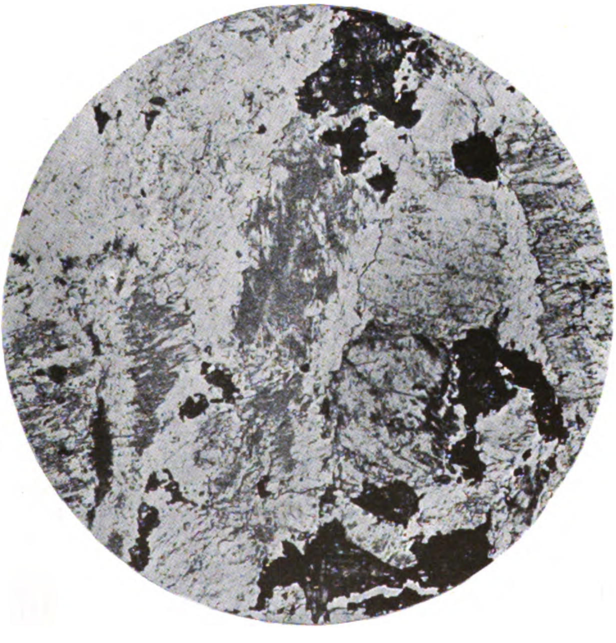

FIG. 2.—SYENITE.

U. S. DEPARTMENT OF AGRICULTURE,

BUREAU OF CHEMISTRY—BULLETIN NO. 80.

H. W. WILEY, Chief of Bureau.

ADULTERATED DRUGS AND CHEMICALS.

I. **Inferior Drugs and Insidious Methods of Deception.**
II. **Rose Geranium Oil and Its Substitutes.**
III. **Phenacetin: Methods of Analysis and Commercial Status.**

By LYMAN F. KEBLER,
Chief of Drug Laboratory.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1904.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., November 6, 1903.

SIR: I have the honor to transmit for your inspection and approval a manuscript reporting the results of investigations relative to the adulteration of certain drugs and chemicals. These investigations were carried on chiefly in the drug laboratory of this Bureau, although some of the work described was done by Mr. Kebler before taking charge of that laboratory. I recommend that this manuscript be published as Bulletin No. 80 of this Bureau.

Respectfully,

H. W. WILEY, *Chief.*

Hon. JAMES WILSON,
Secretary of Agriculture.

INTRODUCTION.

The first two articles of this bulletin set forth the conditions that prevail not only in relation to individuals, but also in some of the best-regulated laboratories. There is a continual cry for cheaper drugs, and in the effort to meet this demand and at the same time make a profit adulteration has spread. The members of the pharmaceutical profession of high standing, however, are anxious to remove from the trade any odium due to adulteration which at present exists.

The third paper deals with the patented medicinal remedy phenacetin, which according to reports has been largely adulterated in this country and many substitutes offered therefor. This subject was studied because of the great interest that exists concerning it in both the medical and the pharmaceutical world, many druggists and physicians being directly involved in the controversy. The fact that phenacetin is sold for 15 cents an ounce in Canada, while $1 or more is charged for the same amount in the United States, creates an impression, correct or incorrect, of injustice. The conditions set forth concerning phenacetin are, moreover, typical of those affecting a large number of patented medicinal remedies. Furthermore, the attempts that have been made to secure such changes in the patent laws as would eliminate these disturbing factors have not been successful. It is hoped that the contents of this paper will place the whole situation before the public in a just and impartial manner.

H. W. Wiley,
Chief of Bureau.

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ADULTERATED DRUGS AND CHEMICALS.

I.—INFERIOR DRUGS AND INSIDIOUS METHODS OF DECEPTION.

INTRODUCTION.

The adulteration of medicinal remedies is brought to our attention from time to time, and it will probably not be denied by many that the basic facts as given to the public are, in the main, correct; but frequently the worst possible interpretation is given them. There are, undoubtedly, members of the pharmaceutical profession whose sense of honor is so distorted by rapacity and greed that they unconsciously, if not deliberately, drift into mendaciousness. These are the men who usually bring the craft into ill repute. Some of the excuses given by these dealers, when informed concerning the shortcomings of their goods, are both interesting and instructive. For example, they contend that it would not be safe to supply their customers with laudanum of full pharmacopœial strength, having in former days used a weaker preparation. Then again, some articles must be modified to suit the tastes of the public, for example, by imparting a certain color to a given preparation. Most of these excuses must be taken *cum grano salis*, but there are undoubtedly adulterations of long standing, such as "limed nutmegs," "bleached ginger," etc., that should be leniently dealt with at present, for it is well known that according to certain ancient methods of curing still practiced the kernels of nutmegs and the rhizomes of ginger are treated with lime to ward off the ravages of insects. Every possible effort should be made to have goods of this character labeled so as not to mislead the consumer. The great difficulty in making any concession whatever is that it might be construed as excusing the dealer's actions and thus embolden him in his insidious sophistry.

When the physician is called to guide a patient through an illness his therapeutic knowledge is generally called into play, and he prescribes for the sufferer on the basis of pure drugs. If he is deceived in the quality of the agents delivered and administered, abnormal symptoms may arise for which the doctor is unable to account, and consequently he is placed in a very embarrassing position. As can readily be seen, most disastrous results may ensue, for which the apothecary is responsible. Many a physician realizes the gravity of the situation, and in self-defense and for the welfare of his patients he prescribes certain brands of recognized purity or carries and prescribes his own remedies, made for him by reputable firms, rather than trust the local druggist with the filling of his prescriptions.

The joint committee of the American Medical Association and the American Pharmaceutical Association, appointed to consider the feasibility of establishing a national bureau of medicines and foods, in its report to the latter body recommended the adoption of the following preamble and resolutions:

> Whereas the foods and medicines supplied in the United States do not so uniformly agree with proper standards of purity, quality, and strength as they should; and
>
> Whereas a degree of distrust and want of confidence concerning the quality of such foods and medicines prevails to a discouraging extent: Therefore it is
>
> *Resolved,* That a more perfectly organized system for remedying the above-mentioned conditions than that now existing should be devised and put into operation; and
>
> *Resolved,* That the American Pharmaceutical Association and the American Medical Association, acting in harmony with the United States Government authorities, constitute the most competent and trustworthy means for obtaining the object named.

Only two of the resolutions are quoted, but they serve to show in a concise manner the present condition of affairs.

During recent years Federal and State authorities have enacted laws which if properly amplified and conscientiously and intelligently enforced will in due time minimize the adulteration of medicinal agents and improve the quality of chemicals. These statutes generally recognize that there are numerous methods by which the quality of a commodity may be impaired. The object of this paper is to discuss some of these methods, cite examples taken from actual practice, and call the attention of the pharmaceutical and chemical world to the various forms of adulteration, which may be subdivided as follows: (1) Conventional, to suit the tastes and demands of the public; (2) accidental or incidental, arising from environment, carelessness, or incompetence on the part of the producer or manufacturer or his agents; (3) arbitrary, to comply with or take advantage of certain fixed, arbitrary standards; and (4) intentional, for gainful purposes and competition.

CONVENTIONAL ADULTERATIONS.

Conventional adulterations, such as silvered cochineal, bleached ginger, and the artificial coloring of many products, have been brought about in various ways. The original object in many cases undoubtedly was to trade on the credulity of the public, for it is a well-known fact that an attractive physical appearance is a great factor in the sale of goods, and color usually carries with it an idea of strength and quality. Such a firm footing have these factors secured that in some cases the adulterated article is selected rather than the pure. To illustrate, a few years ago, while exhibiting a sample each of pure and weighted cochineal, a recent graduate of one of our well-known colleges, after looking them over thoroughly, selected the adulterated article as the genuine product. When informed of his error, somewhat chagrined at his mistake, he said, "It is the only kind I have ever seen and of course I thought it was the pure article." From this and similar experiences it would seem that some of our educational institutions are not exercising as much care as they might in the selection of their material for instruction purposes.

The artificial coloring of preparations is the most widespread of conventional adulterations. These pages will probably not come under the eyes of a single reader who is unable to enumerate a score of such products. To suggest a multitude of these goods it is only necessary to enumerate elixirs, tinctures, sirups, essences, pills, and tablets. As long as the harmless vegetable colors were used little cause for anxiety existed, but of late the danger line has been passed by the extensive use of aniline dyes. The finding of harmful coloring agents in food products is a matter of common repute, and medicines are not exempt. It would probably not be desirable to interdict the use of harmless coloring agents, but their use should be discouraged. In cases where it is clearly evident that fraud is concealed by coloring agents, as in the preparation of vanilla extract from the chemical vanillin, dissolved in a suitable menstruum and colored with caramel, a suitable punishment should be provided.

It is quite possible that an attractively colored preparation is more palatable than one that is not colored, but the nature of the coloring agent should, in such case, be plainly set forth on the label, unless it is part of a formula contained in some recognized authority like the Pharmacopœia or the National Formulary.

BLEACHED GINGER.

The bleaching of ginger by covering the fingers with some white substance, like calcium carbonate, is frequently done to cater to the fancy of certain customers. It also seems to be incidental to certain processes of curing. In some cases, however, the prime motive is to conceal inferiority. The amount of coating added usually does not exceed 5 per cent, but the increase in price is about 15 per cent. In bleaching ginger considerable care must be exercised in the selection of the fingers, or the shrinkage due to loss of moisture will be equal to the amount of weighting material added. At all events the purchaser of bleached ginger pays more money and gets less ginger than if he purchased the unbleached variety.

COCHINEAL.

Availability of the Pure Product.

It seems that the cochineal industry is grossly involved in adulteration, and, according to some dealers, irretrievably lost to this practice. They contend that it is almost impossible to purchase a pure article on the open market, but an investigation shows that this contention is not well founded. In fact, the pure product is so easily secured that adulterated cochineal would be placed under the fraudulent form of adulteration if it were not well known that the consumer virtually knows only the silvered or commonly adulterated variety. The object of the work done on cochineal was to ascertain, not the extent of the adulteration, but how difficult it is to obtain the pure article. It was soon found that pure cochineal was readily available when demanded. A number of samples examined gave the following results:

Analyses of cochineal.

Serial No.	Source.	Kind.	Ash.	Adulterant.	Moisture at 105° C.	Coloring matter.		Price per pound.
						Colorimetric.	1 per cent solution of K_3FeCy_6.	
			Per ct.		*Per ct.*		*cc.*	
3	....	Silver	30.81	Talcum	6.7	64	1.2	$0.40
156	Eimer & Amend	Powdered	17.25	Earthy matter.	6.25	50	1.0	.45
157	Smith, Kline & French Co.	Black	4.94	None	7.00	100	1.8	.45
158	do	Powdered	6.00	do	6.15	100	2.0	.50
159	do	Silver	3.01	do	6.38	92	1.3	.40
165	Eimer & Amend	Black	4.71	do	7.73	100	1.9	....

Nos. 3 and 156 represent goods delivered to the Bureau of Chemistry without specification. No. 159 was sent with the belief that it was adulterated. Nos. 157, 158, and 165 were delivered as pure goods, and an examination proved such to be the case. On account of the high price, the variable quality, and the customary adulteration of cochineal, suitable methods for readily determining its quality are very desirable.

Methods of Analysis.

The per cent of ash is a fair index in determining the purity of cochineal, but this factor can easily be circumvented by the addition of starch, mixing exhausted material with the pure article, or molding paste into small grains to resemble closely the outlines of the insects themselves. The ash of normal cochineal should never exceed 6 per cent.

The most reliable procedure to determine the quality of cochineal is to estimate the amount of carmine either colorimetrically or by one of the oxidation methods. J. Löwenthal's[a] well-known method for the determination of tannin can be employed to advantage only when considerable work of this character is done. F. Penny's[a] process appears to have many advocates. The basis of this method is the oxidation of the coloring material in an alkaline solution by means of a 1 per cent solution of potassium ferricyanid. It is executed by digesting together, on a water bath, for one hour, 1 gram of powdered cochineal and 5 grams of caustic potash, dissolved in 20 cc of distilled water. Avoid dissipation of the water, dilute the resulting mixture to 100 cc, and titrate an aliquot part by means of the potassium ferricyanid solution. The carmine red color is changed to a brownish yellow. The transition of color is indistinct and the exact end reaction is difficult to determine. In this work 20 cc of cochineal solution were used for each titration.

---

[a] Ztsch. anal. Chem., 1877, 16: 179.

Except in cases where cochineal is to be used for special purposes, a simple colorimetric method gives satisfactory results. For this purpose the following process is suggested:

Digest on the water bath for one hour, 1 gram of powdered cochineal and 1 gram of potassium hydroxid, dissolved in 20 cc of water; replenish the water as it evaporates and make the mixture up to 100 cc with distilled water. Dilute 10 cc of this solution to 400 cc. The color thus obtained, from a cochineal of known purity, is taken as the basis and called 100. If pure cochineal always possessed the same tinctorial value and were a well-known commercial article, the color obtained by the above procedure could be utilized as a standard. A readily available uniform standard is, however, found in a properly diluted aqueous solution of potassium permanganate. It has been found that by diluting 12.5 cc of a centinormal potassium permanganate solution (0.316 gram of pure potassium permanganate dissolved in 1 liter of distilled water) to 100 cc, a tint of color is obtained, when observed in a Nessler tube held at a right angle to the eye of the worker, which is identical to that prepared from pure cochineal by the process described above. By adopting this potassium permanganate solution as a standard, and calling it 100, the tinctorial value of every sample of cochineal can be ascertained and definitely expressed. If a sample of cochineal should be found in the future possessing a higher coloring equivalent than any met in this work, the standard of comparison would not need to be changed, but could be expressed by 110, 125, etc., as the case may be.

According to the above method, Nos. 157, 158, and 165 are of good and equal quality, No. 159 is of fair quality, and Nos. 3 and 156 are decidedly inferior, having only about one-half and two-thirds, respectively, the coloring value of a normal cochineal. A microscopic examination of No. 159 indicates that the silvering is due to rod-shaped bodies, like bacilli, but the usual bacterial stains would not effect them. It is quite possible that the whitish appearance is due to a dried residue of cryptogamic plant growth. It is interesting to note that the results obtained by the potassium ferricyanid process run nearly parallel with the colorimetric data.

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[a] Rept. Brit. Assoc. Advanc. Science, 1855, pt. 2, p. 68.

The physical appearance of these conventionally adulterated articles has secured such a firm footing in the public mind that it is almost impossible to replace them by pure goods. To eradicate these deeply inculcated erroneous ideas will require years of patient effort by way of both exposure and education.

ACCIDENTAL ADULTERATIONS.

Accidental adulterations are very widespread, and it is frequently difficult to say where this form of adulteration ends and the fraudulent begins. Crude drugs usually contain admixtures of twigs, stems, dirt, foreign leaves, and a host of other bodies. The Pharmacopœia does not make any allowance for contaminations of this kind, but dealers contend that such hypothetical requirements are purely academical and have no place in the commercial world. The argument is also advanced that certain drugs are collected by ignorant, semi-civilized people who can not be expected to eliminate impurities of this character. Excuses of this nature do not in any way relieve dealers and manufacturers from their responsibilities to the public. It is plainly their duty to handle and use only goods of the proper quality. To what extent some of these foreign articles modify the primary action of a drug can not be conjectured.

A certain few of the large drug houses of this country are eliminating these impurities by garbling. When it is remembered that the foreign material frequently amounts to 20 per cent or more it is quite evident that the cost of garbled goods is materially increased and that dealers in clean drugs are distinctly handicapped when brought into competition with those who handle inferior grades. It would probably be unjust to request a complete absence of foreign material, but a maximum limit could readily be fixed. A concerted effort should be made by all large manufacturers to establish a uniform high standard for all drugs used by them either in the making of finished medicinal remedies or for powdering. All purchases should be made on the basis of an adopted standard, paying only for the actual amount of good material in a consignment.

By a recent act of Congress the Secretary of Agriculture is authorized to investigate the quality of drugs imported into this country. The drug laboratory has already taken steps toward the securing of samples, which will be carefully investigated, in cooperation with the Bureau of Plant Industry.

It would probably be too exacting to require a root to be freed from all extraneous matter, but an upper limit of ash should certainly be fixed. Some time ago a sample of golden seal root was received which was intended for powdering; it contained 23.8 per cent of ash and 2.02 per cent of hydrastine alkaloid, based on the air-dried material. A normal root should not contain more than 10 per cent of ash and not less than 2.5 per cent of hydrastine alkaloid. With golden seal at 60 cents per pound this dirt is a profitable addition for someone.

Certain leaves almost always contain a considerable amount of foreign matter. Chimaphilla leaves have been seen which were mixed with 25 per cent of stems. A sample of jaborandi leaves recently examined contained not less than 16 per cent of twigs and stems. A coca leaf sample on assay indicated 0.52 per cent of cocaine alkaloid, but the leaf was mixed with at least 18 per cent of foreign material. No valid excuse exists for this evil. Coca leaves containing as little as 3 per cent of foreign matter are readily available. A sample of cubeb berries on examination gave the following results: Stems, 15 per cent; worthless berries, 11 per cent; oil, 6.38 per cent. The physical appearance of the oil was good, its specific gravity 0.9384, and optical rotation −34.6°. The gravity is a little high but can hardly be considered abnormal. Good berries should yield not less than 12 per cent of oil.

DETERIORATED DRUGS.

The sample of cubeb berries referred to serves as an excellent example of transition between drugs containing foreign admixtures and those that have deteriorated by age or manner of keeping. To what extent these cheap, inferior, and in some cases worthless goods are used it is difficult to ascertain, but from information vouchsafed by brokers, drug millers, and manufacturers, this practice obtains to a considerable extent all about us. Deteriorations are incidental to the drug business, but the use of such goods, knowingly, in the manner indicated, is fraudulent, and what makes this practice so extremely reprehensible is the fact that it is very difficult, if not impossible, to detect inferior material of this character when powdered with goods of prime quality.

Articles particularly susceptible to change due to time are those containing essential oils, such as cinnamon bark, clove buds, lavender flowers, peppermint herb, sandal-wood chips, etc. A hundred-pound package of cinnamon-bark chips when submitted to distillation yielded only enough oil to impart a distinct flavor of cinnamon to 5 gallons of the aqueous distillate. Potent drugs with only a trace of alkaloids, due to old age, improper collection, or damage in transportation, are frequently used. Old jaborandi leaves deficient in alkaloidal strength are met with at times. Belladonna leaves improperly collected are not uncommon, and damaged coca leaves are occasionally placed on the market.

"C. P." CHEMICALS.

No less an authority than E. W. Morley during an interview said: "It is virtually impossible to make a chemical absolutely 100 per cent pure." From this it would seem that we must expect to find a small amount of foreign material in all chemicals. Experience shows such to be the case. These impurities are usually incidental to the process of manufacture, but when the amounts are excessive it must be ascribed to either carelessness, ignorance, or a desire on the part of the manufacturer to prepare a cheaper article at the expense of purity.

Chemical reagents are usually supplied as being chemically pure, "C. P." This designation was formerly supposed to mean, as it now should, a chemical of a high degree of purity. Whether this quality of chemicals was ever of a higher degree of purity than at present is difficult to ascertain. Occasionally we find a note in chemical literature to the effect that certain chemicals represented as free from impurities contained appreciable quantities of foreign matter. For example, Classen calls attention to two samples of "chemically pure" bismuth, sold for "scientific purposes," one of which contained 2 per cent of lead and the other 1.5 per cent of copper and 0.5 per cent of iron. Every sample of bismuth ever examined by the writer contained traces of arsenic and iron. Notwithstanding the fact that great improvements have been made in the manufacture of chemicals within recent years, the stage has not yet been reached in which chemicals are absolutely free from all impurities. It is true some of them will indicate a purity of 100 per cent, but this is usually due to the impurities in one chemical offsetting those in another, or to limitations of analytical methods. For example, recently a purchase of high-grade potassium permanganate was tested and found to analyze 100 per cent pure, and yet the chemical contained traces of nitrates and chlorids. The oxalic acid solution was prepared from oxalic acid that had been recrystallized in the laboratory with every possible precaution, and a careful examination showed that it was free from all impurities ascertainable by chemical analysis. Furthermore, the normal solution of oxalic acid was tested as to strength by the best methods and found to be correct.

The designation "C. P." as used at present is not only meaningless and worthless, but misleading in the extreme. Chemicals of the poorest character are marked "C. P." Manufacturers use the term carelessly, and dealers will attach it to any article of a chemical nature that they think will thus be made more attractive. Such dealings are an imposition on the consumer and unfair to honest competitors. Some time ago an examination was made of sodium carbonate crystals, C. P., delivered on a competitive bid, which were found to be about equal in quality to commercial English sal soda. Circumstantial evidence pointed strongly to the conclusion that this dealer had simply marked the English brand with the classical abbreviation "C. P." and delivered it as such. Again, the writer has known of carloads of C. P. glycerin, shipped in iron drums, that contained a sediment consisting of iron scales, fibrous material, and other débris. These goods were also delivered on contract, awarded on sample submitted and competitive bid. If these consignments had not been examined on delivery they would have been accepted without question, but after their true nature became known they were promptly rejected. Honest dealers handling goods of prime quality can not successfully compete with articles of this character unless each consignment is examined.

A few more cases will be sufficient to show the quality of some of the C. P. chemicals frequently delivered. C. P. glycerin often contains arsenic and certain bodies that reduce an alkaline copper solution, and invariably contains volatile fatty acids. One sample of potassium iodid, C. P., was found to be contaminated with sulphates, iodates, and sodium compounds. It also contained 1.5 per cent of chlorid, and 5 grams required 3 cc of decinormal acid to neutralize the alkalinity. A sample of potassium bisulphate C. P. contained only 33 per cent of the acid sulphate. Some calcium oxid C. P. was found to contain iron, aluminum, magnesium, sulphate, and siliceous matter, being virtually no better than ordinary commercial quicklime. A sample of calcium chlorid C. P. was found to be contaminated with iron, aluminum, and magnesium, and was highly alkaline to litmus paper. Such articles would certainly be objectionable for analytical work.

Certain dealers, laboring under the delusion of hypercritical standards, have sought shelter behind the very elastic term "pure" and are supplying the market with chemicals which they think are sufficiently pure for all practical purposes. The term "pure" conveys distinctly different meanings to the artisan, assayer, broker, chemist, manufacturer, photographer, physician, and toxicologist. As a rule, these consumers do not call for chemicals free from all conceivable impurities, but demand the absence of certain specified contaminations which are detrimental in special work. The toxicologist must have his zinc free from arsenic, phosphorus, antimony, and sulphur, while traces of copper, carbon, or lead may not be objectionable.

There are a number of other terms used which at present convey very little information; they are Purum, Purissimum, Purified, Twice Purified, and Absolutely Chemically Pure; and even U. S. P., Br. P., and Ph. Ger. IV, are indifferently employed. Within recent years manufacturers have adopted a custom of marking some of their chemically pure chemicals "Free from manganese," "Free from sulphur," "Free from iron," "Arsenic free," "Free from silver," and "Strictly chemically pure, free from N. and S." The marking of chemicals as free from certain impurities is certainly a step in the right direction, but the great difficulty is that many of these chemicals are free from these impurities only on the label. A recent consignment of copper sulphate to be used in sugar analysis, delivered to the Bureau of Chemistry on the specification that it must be strictly free from iron, was so labeled, but nevertheless contained this impurity in appreciable quantities. A purchase of magnesium oxid guaranteed to be free from sulphur and so labeled contained over 2 per cent of sulphur calculated as anhydrous sodium sulphate, and goods labeled "Chemically pure sulphuric acid, arsenic free," frequently contain this impurity.

In view of the above facts it is quite evident, first, that "C. P," or "Chemically pure," with all its qualifying adjectives, at present means nothing, and its fraudulent use should be prohibited; second, that no chemical should be accepted as free from a certain impurity simply because the package is so labeled; and, third, that certain specific standards for chemical reagents should be established. Such reagents should be free from all undesirable or detrimental contaminations, and this fact should be set forth on the label. It is well known that many original packages are not marked at all except by the private mark of the manufacturer. A Federal law requiring that every package of chemicals be properly labeled as to name and quality would remedy this practice. V. Coblentz, in this connection, well says: "The indiscriminate labeling of chemicals without qualification as to degree of purity should by all means be discouraged as being a loose practice through which legal responsibilities may be evaded."

ADULTERATIONS RESULTING FROM ARBITRARY STANDARDS.

The argument is occasionally advanced that arbitrary standards are direct incentives to fraudulent dealings. Fluid extract of nux vomica prepared from a bean containing 2.5 per cent of total alkaloids is diluted, to conform to a standard, so that it contains only 1.5 per cent of alkaloids. Milk dealers reduce milk containing 5 per cent of fat, so as to pass a 3 per cent standard. The former is considered right, the latter reprehensible to a high degree. In reality the one does not appear to be any worse than the other.

The United States Pharmacopœia prescribes an upper and a lower limit of morphine for powdered opium, but no provisions are made to reduce an opium containing more than 15 per cent of morphine, the highest amount permissible, so as to conform to the proper requirements. It is not uncommon to meet with powdered opium containing over 15 per cent of morphine, and dealers are compelled in self-defense to reduce it to the proper strength by mixing it with opium of a lower grade, or to use it in the manufacture of other medicinal remedies containing morphine. If the manufacturer places powdered opium on the market containing more than 15 per cent of morphine, he becomes liable to punishment, and if he dilutes it to the proper strength with a detectable diluent many State laws consider him culpable. In fixing standards of the above type it is desirable to provide for contingencies of this character.

Some dealers maintain that the pharmacopœial requirements of certain oils are abnormal and that adulteration must be resorted to if oils of the desired quality are supplied. Much capital is made of this in certain quarters, by quoting such oils as bay, coriander, and pimento, "compounded to conform to the requirements of U. S. P. 1890," at from 30 to 60 per cent below the price asked for pure oils which are not expected to comply with the pharmacopœial standards.

ANALYSES OF CERTAIN OILS AND POTASSIUM CYANIDS.

The most important factor in judging the quality of oil of bay, aside from its peculiar odor, is the specific gravity, which should lie, according to the Pharmacopœia, between 0.975 and 0.990, at 15° C. An examination of ten samples of pure oil of bay, obtained directly from the distillers, gave specific gravities varying from 0.958 to 0.980. All but two fell below the lower limit. Other recognized authorities allow a lower limit of 0.965. Three of the above samples fell below this standard, being 0.958, 0.9627, and 0.964, respectively. On submitting 511 pounds of bay leaves to steam distillation, 12.5 pounds of light and heavy oils mixed were obtained, which after aging one year had a specific gravity of 0.955. It would therefore seem that the specific gravity of the Pharmacopœia for oil of bay is a little too high.

Six samples of coriander-seed oil were examined, of which three were marked pure, two were distilled by the writer, and all complied with the U. S. P. requirements. The sixth was marked "German," and proved to be adulterated. The pharmacopœial standard is therefore not far from the truth.

Six samples of oil of pimento berries were tested. Their specific gravities were as follows: 1.0494, 1.0510, 1.0280, 1.034, 1.040, and 1.035. In other respects these oils complied with the pharmacopœial requirements. The first two were labeled, "Made to conform to the U. S. P. requirements." The last was distilled by the writer, and the others were secured from prominent distillers in this country and guaranteed pure. The Pharmacopœia requires the specific gravity to fall between 1.045 and 1.055, but "Die Aetherische Oele," by Gildemeister and Hoffmann, recognizes a specific gravity as low as 1.024. While the specific gravity is, to a certain extent, an indication as to the amount of eugenol present, it would not be wise, in view of the above facts, to pronounce an oil adulterated simply because it had a specific gravity below 1.045.

Potassium cyanid 98 to 100 per cent pure has assumed considerable commercial importance. In ordering this article it is customary to specify the per cent of cyanid only. For financial and technical reasons potassium cyanid is largely mixed with sodium cyanid. There can be no real objection to this practice, if the goods are properly represented, but a mixture of these two cyanids should not be delivered for 98 to 100 per cent potassium cyanid. In determining the percentage of potassium cyanid in a mixture of this character the results will be above 100 per cent in proportion to the amount of sodium cyanid present. In order to meet this difficulty manufacturers add, or do not remove, certain inert substances, which usually consist of carbonates, chlorids, or mixtures of both.

An examination of four samples of potassium cyanid, labeled 98 to 100 per cent pure, gave the following results:

Analyses of 98 to 100 per cent potassium cyanid.

Sample No.	Cyanid (calculated as KCy).	Potassium cyanid.	Sodium cyanid.	Potassium carbonate, etc. (by difference).	Moisture at 125° C.	Sodium chlorid.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	101	33.65	50.56	0.70	0.82	14.27
2	111.19	13.62	73.65	12.82	....	....
3	105.87	31.75	55.93	12.32	....	....
4	95.33	65.90	22.20	8.82	1.58	1.66

All of the above samples represent imported goods. The results are self-explanatory. No valid reason for the practice of mixing the cyanids in the manner shown and selling the mixture as potassium cyanid has thus far been offered by manufacturers.

METHODS OF ESTIMATING CHLORIDS IN SOLUBLE CYANIDS.

The present methods for detecting and estimating chlorids in the soluble cyanids are not satisfactory. It is usually stated that silver cyanid is soluble in concentrated nitric acid, whereas silver chlorid is insoluble, but in practice it is not safe to rely on this test. The fusion method is useful but dangerous. It consists in heating to redness an intimate mixture of 1 gram each of the cyanid and potassium nitrate and 5 grams of potassium carbonate. The carbonate and nitrate must be free from chlorids. In this mixture an extremely efficient oxidizing agent and one of the best-known reducing agents are brought together. When the mixture is fused, there is a more or less vigorous chemical reaction, and frequently an explosion results. A more expeditious and satisfactory method would therefore be welcome.

The gravimetric method on the next page is based on the fact that cyanids reduce permanganates.

In a 250 cc beaker, containing 150 cc of water, dissolve approximately 1 gram (accurately weighed) of the cyanid. Heat to boiling, then cautiously add about 2 grams of potassium permanganate or such a quantity that the solution after having been heated for one-half hour on the steam bath is still distinctly pink. Destroy the excess of potassium permanganate with oxalic acid, heat the mixture a few minutes on the water bath, and the precipitate will subside, leaving a colorless layer of liquid. Filter the solution hot, wash the precipitate on the filter paper with 200 cc of hot water, mix the filtrates, render distinctly acid with nitric acid, and heat the mixture to boiling. In this solution the chlorids are estimated in the customary manner.

Mr. J. K. Haywood, chief of the insecticide and agricultural water laboratory of the Bureau of Chemistry, employs a volumetric method with satisfactory results. It has been carefully compared with the above gravimetric process and found to accord with it uniformly. The method is as follows:

Weigh accurately in a weighing bottle about 10 grams of the substance, dissolve in water, and make up accurately to 1 liter. In an aliquot part of this solution estimate the cyanogen by titrating with a decinormal silver nitrate solution. The end reaction is shown by the appearance of a white cloudiness in the solution. Now add potassium chromate, as indicator, and titrate with the standard solution of silver nitrate until the appearance of the usual reddish-brown color of silver chromate.

The reactions involved in the above method are represented by the following equations:

(1) $AgNO_3+2KCN=AgCNKCN+KNO_3$.
(2) $AgCNKCN+AgNO_3=2AgCN+KNO_3$.
(3) $NaCl+AgNO_3=AgCl+NaNO_3$.

Representing the number of cubic centimeters of silver nitrate solution used in the first titration by A and the number of cubic centimeters used in the second titration by B, then B—A equals the number of cubic centimeters of decinormal silver nitrate solution required to combine with the chlorids present, while the amount of cyanogen can be calculated from A, as indicated by the first equation given.

INTENTIONAL ADULTERATIONS.

With very few exceptions the underlying motive of all intentional adulterations is monetary gain. Some of the sophistications considered above appear to have some superficial excuse for existing, but those here enumerated as intentional are deliberately premeditated misrepresentations, and should be dealt with summarily. In this category comes the potassium cyanid reported above. Other instances are: Borax diluted with sodium bicarbonate; cornstarch delivered when St. Vincent arrowroot is asked for; prime quality drugs mixed with inferior or partially exhausted goods; spurious sandalwood oil containing chloroform added to raise the specific gravity and to increase the apparent content of santalol; powdered drugs in the preparation of which inert and deteriorated products have been used; beeswax with its numerous adulterations; turpentine diluted with kerosene, etc.

A sample of beeswax recently examined was found to contain 33 per cent of cassava starch. This variety of starch indicated that the adulteration was of southern origin. On inquiry it was found that about $800 worth of this fraudulent material had passed through the New York custom-house, having been imported from Mexico.

Another sample of beeswax on examination gave the following results: Melting point, 61.5° C.; specific gravity at 15° C., 0.959; acid number, 14.2; ether number, 73.6. These numbers do not materially differ from those usually recognized as normal for pure beeswax, except the acid number, which is a little low. A qualitative examination showed that this article consisted, for the greater part, of a high melting point ceresin and Japan wax, the mixture being probably flavored artificially to resemble the genuine product. New York is the home of the apiary which produced this remarkable beeswax.

The adulteration of beeswax with starch is discouraging, because it shows that the days of gross sophistication are not past; but to find a scientifically prepared mixture closely resembling beeswax is most deplorable, because it indicates that men of education are using their talents to serve a dishonest purpose.

The drug laboratory has in its possession a small amount of a flavoring agent, which has been exploited as of much service in beeswax adulteration, but the nature of its composition has not as yet been determined.

Twenty-five per cent of all turpentine, as usually purchased in small packages, is liberally adulterated with kerosene. The present analytical methods do not appear to be sufficiently refined to detect this adulterant with certainty when present in small quantities. Investigations are under way at present which it is hoped will remedy this defect.

SIFTINGS AND SWEEPINGS.

In the handling of drugs more or less of the finer particles escape from the bales, and in the larger warehouses the practice has been established of collecting this material from the floors, as occasion requires, and offering it to the trade at a low figure. The conditions under which these sweepings and siftings are accumulated and collected naturally lead to the belief that they are likely to be contaminated to a considerable degree. Products of the above character commonly met with are the cinchona barks, cochineal dust, pepper, tea, and senna leaves.

Some of the sweepings and siftings are utilized in extracting certain active principles, like caffeine from tea sweepings, and certainly nothing could be said against an economy of this kind, but the practice of using articles of this character in the manufacture of medicinal preparations should certainly be discountenanced. Some manufacturers frankly admit that this practice obtains in their works, but maintain that these goods are "just as good" as those for which a high price is paid.

Three bales of calisaya bark siftings, all containing foreign material, on examination gave the following results: The per cent of total alkaloids was 0.47, 3.6, and 4.7. The per cent of ether-soluble alkaloids was not determined in the first bale recorded, and was 1.9 and 2.7, respectively, in the last two. A good calisaya bark should not contain less than 6 per cent of total alkaloids nor less than 3.5 per cent of ether-soluble alkaloids.

Every bale of senna siftings and every bag of tea sweepings examined contained a goodly proportion of extraneous matter. Broken senna leaves are usually of good quality.

A COMMON METHOD OF DECEPTION.

A very significant editorial appeared in the British Food Journal,[a] from which the following extract is taken:

> The substitution of an imitation of some kind for the article actually asked for or desired by the purchaser is a particularly mean form of deception which is practiced nowadays to an almost incredible extent. It is astonishing and mournful that so many persons should be concerned in the initiation, fostering, and carrying on of so shameful a system, and that others are found who in speech and print seem willing to lend to it either their countenance or condonation.

Still more reprehensible is the practice of submitting a sample of prime quality and then, on receipt of an order, delivering goods of an inferior grade.

Some may think that the above method of deception does not obtain to any extent, but only a superficial investigation will show that it permeates many lines of business, and those who are responsible for the quality of the medicinal remedies supplied the unfortunate sick should ever exercise eternal vigilance. The following examples, which are typical of this practice, should serve to banish the remotest doubt. A sample of belladonna leaves contained by acid titration 0.438 per cent of total alkaloids. On receipt of the consignment delivered as per sample submitted, nine bales were tested with the following results: 0.12, 0.14, 0.11, 0.10, 0.13, 0.12, 0.13, 0.30, 0.11 per cent of total alkaloids. A sample of potassium bromid complied with U. S. P., 1890, requirements. The goods supplied were inferior in every respect. A podophyllin sample was of U. S. P. quality, but the article delivered contained much material insoluble in alcohol. A linseed-meal sample contained 33 per cent of pure oil. After placing an order on the strength of the sample, two carloads were delivered, which, on examination, proved to contain on the average 35 per cent of oil, having the following properties: Specific gravity at 15° C., 0.9055; acid number, 6; saponification number, 99.7. The oil extracted from the linseed meal was highly adulterated with mineral oil, which was added to the flaxseed meal after a part of the natural oil had been expressed. A sample of oil of wormwood of good quality was submitted, but the goods delivered were adulterated with turpentine. A sample of tea sweepings submitted contained 2.64 per cent of caffeine alkaloid. A ton subsequently delivered contained only 1.6 per cent of this alkaloid. A sample of caramel possessed a coloring equivalent of 100, and gave good tests in every respect. On examining a delivery of about 5,000 pounds it was found to have a coloring equivalent of only 80, and deported itself badly in every way. These examples could be largely extended, but the above fully illustrate existing conditions.

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[a] 1903, vol. 5, p. 97.

It will be observed that most of the examples cited represent the larger transactions and usually involve manufacturers and producers. Many members of the American Pharmaceutical Association and the National Wholesale Druggists' Association, and some of the best informed manufacturers and jobbers, have placed themselves on record as conversant with these undesirable methods and anxious to prevent their use, but they are greatly handicapped by competition. Such men hope to see the day when all druggists, retail or wholesale, will be compelled by some competent authority to deal only in high-class medicinal agents.

II.—ROSE GERANIUM OIL AND ITS SUBSTITUTES.

In spite of the great advance made in the chemistry of essential oils during the past decade, ample evidence can readily be collected to show that this is as yet a fertile field for the adulterator. Many kinds of manipulators are found, from the tyro who endeavors to palm off oil of French turpentine for oil of rue, and the distiller who sprinkles his rose leaves with geranium oil before distilling, to the chemist who is an abettor to the use of acetin and glycerin in volatile oils for the purpose of increasing the apparent content of ester and alcohol, respectively.

By referring to the various price lists it will be found that the quotations for the geranium oils vary from $12 per pound for the Spanish to $2.25 for the Turkish oil; and ginger-grass oil, which is conceded to be only another name for an inferior Turkish oil, sometimes highly adulterated, is quoted at $1.10 per pound. Certainly here seems to be a great opportunity for the clever manipulator, and aside from the assistance of a well-trained nasal organ, let us see what are the probabilities of detecting such adulteration.

Rose geranium oil is a colorless, yellowish, greenish, or brownish liquid, depending on the manner of distillation and storage, and has a pleasant rose-like odor. Its specific gravity varies from 0.8878 to 0.9073; optical rotation in a 100 mm tube, -6° to -16°; ester, calculated as geranyl tiglinate, varies from 8 to 42 per cent. All varieties are soluble in 2 to 3 volumes of 70 per cent alcohol, except the Spanish, which is rendered turbid by the presence of a small amount of separated paraffin. The chief constituents are geraniol and citronellol, the total content of which, both free and combined, varies from 60 to 85 per cent. The former usually exists in much the greater proportion.

Turkish or Indian geranium oil, also known as palma rosa, Indian grass oil, and rusa oil, usually closely resembles rose geranium oil in physical appearance, solubility, specific gravity, and percentage content of alcohols and esters. In odor there is frequently a close resemblance and the optical rotation varies from $+2^\circ$ to -2°.

Ginger-grass oil is supposed to be an inferior quality of palma rosa, and its properties, therefore, should very closely resemble those of the latter oil, excepting possibly its odor, unless it is highly diluted with turpentine or mineral oil, as is frequently the case.

It can readily be seen that a judicious mixer could combine oils possessing the properties described above so as to bewilder a chemist, even though he were well versed in the chemistry of essential oils.

Jeancard and Satie[a] have studied these oils to some extent, and think they can distinguish between them by their contents. The following table is taken from their work:

Analytical data on pure geranium oils.

Origin.	Density at 15° C.	Rotation at 15° C. in 100 mm.	Saponification value.	Esters.	Alcohols.
		°		*Per cent.*	*Per cent.*
Cannes	0.8972	−9.40	54.60	9.80	66.31
Spain	.9073	−7.30	65.80	7.84	66.23
Corsica	.9012	−8.00	60.20	7.00	68.55
Africa	.9006	−8.06	65.80	8.08	63.19
Bourbon	.8905	−8.20	74.00	6.65	71.28
India	.8960	− .48	43.00	11.30	84.62

The per cent of esters and alcohols is based on the formulæ $C_{12}H_{20}O_2$ and $C_{10}H_{18}O$, respectively.

In view of the fact that the highest-grade oils grow in certain localities only and bring fancy prices, the opinion is ventured that it would not be safe to deduce any general statement from the above results, except in the case of Indian oil.

Some time ago the writer received a number of samples of geranium oils in original packages bearing the labels of the largest and best-known essential-oil dealers in the world, with the request that an opinion be given as to their purity and quality. On submitting the samples to an examination the results tabulated below were obtained:

Analyses of geranium and allied oils.

Kind of oil.	Color.	Specific gravity at 15° C.	Acid number.	Esters as tiglinate.	Alcohols.	Opt. rot. in 100 mm tube at 20° C.	Solubility in 70 per cent alcohol.
				Per ct.	*Per ct.*	*Degrees.*	
African (1)	Greenish yellow	0.8901	4.51	20.48	76.06	− 6.73	Soluble in 2 volumes.
African (2)	Lemon yellow	.8915	6.00	28.95	63.49	− 8.41	Insoluble in 10 volumes.
African (3)	Orange yellow	.9053	6.09	18.95	42.34	−19.20	Do.
African (4)	Lemon yellow	.9319	4.47	95.00 [a]78.85	71.08	− 7.70	Soluble in 1½ volumes.
Algerian (1)	do	.8981	3.20	83.61	65.57	−17.00	Insoluble in 10 volumes.
Algerian (2)	Colorless	.8961	2.87	36.46	73.14	− 9.60	Soluble in 2½ volumes.
Reunion	Lemon yellow	.8878	6.28	28.89	72.13	− 7.66	Soluble in 2 volumes.
Palma rosa	Orange yellow	.8968	1.80	22.30 [a]18.50	81.75	− 2.90	Do.
Turkish (1)	Lemon yellow	.9142	4.13	12.13 [a]10.06	50.00	−50.70	Insoluble in 10 volumes.
Turkish (2)	do	.9154	2.41	14.25 [a]11.72	72.93	+ 1.91	Soluble in 2 volumes.
Ginger grass (1)	do	.9213	1.50	8.07 [a]6.69	20.18	−69.13	Insoluble in 10 volumes.
Ginger grass (2)	do	.9234	3.88	1.46 [a]1.20	21.77	−65.00	Do.
Rhodonol II	Colorless	.8758	......	9.00 [a]7.42	......	−26.9	Soluble in 2 volumes; insoluble in more.

[a] Per cent of ester calculated as geranyl acetate.

The acid number was determined by dissolving a given weight of the oil in strong alcohol, in which all the oils were soluble in all proportions, and titrating with decinormal alcoholic potash, at the ordinary temperature, using phenolphthalein as indicator. The figures indicate the number of milligrams of potassium hydroxid required to neutralize the acidity of one gram of oil. The esters were estimated by adding an excess of alcoholic potash to the above solution, heating to boiling with a reflux condenser for about one hour, then titrating back the excess of alkali by means of decinormal acid. From the amount of alkali consumed the necessary calculations can readily be made, either as geranyl tiglinate ($C_{10}H_{17}CO_2C_4H_7$) or geranyl acetate ($C_{10}H_{17}CO_2CH_3$), as the case may require. The percentage of alcohol, free or combined, was determined by acetylizing a given weight of oil with an equal amount of acetic anhydrid, in the presence of fused sodium acetate. The acetylized product was purified by washing with water, and rendered anhydrous by means of fused sodium sulphate. A given weight of the acetylized oil was then saponified, as outlined above for determining esters, and from the data thus secured the desired calculations were made.

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[a] Bul. Soc. Chim., 1900 (3), **23**: 37.

In computing the amount of alcohol, both free and combined, in geranium oil, it must be remembered that the chief ester of the natural oil is a tiglinate, and on acetylizing with acetic anhydrid the free alcohols are converted into acetic esters. We therefore have a mixture of esters on which to base our calculations. The percentages of alcohols given in the table above were computed from the mixed esters. The question might arise as to whether any of the tiglinic group was replaced by the acetyl group, but this inquiry can not be answered here.

The first African oil, the second Algerian oil, and the Reunion oil are normal in every respect. The African oil No. 2 and the Algerian oil No. 1 can be considered normal except as to solubility, and it is doubtful whether these oils can justly be considered adulterated. The third African oil is low in alcohol content, high in optical rotation, and insoluble in the proper amount of 70 per cent alcohol. The fourth African oil is a spurious product, which did not respond affirmatively for a hydroxyl group when tested by means of acetyl chlorid in the conventional manner, thus indicating the absence of any alcohol. The palma rosa oil and the Turkish oil No. 2 are both normal India products, while the first Turkish oil is abnormal. Both of the ginger-grass oils are entirely different from anything described in literature. Judging from the high specific gravities and high optical rotations, these ginger-grass oils are not adulterated with either turpentine or mineral oil. The last oil named, Rhodonol II, is apparently a fairly pure geraniol.

It is clearly evident from the data obtained in this investigation and other work on record that a scientific adulterator could readily mix some of the cheaper geranium oils with the expensive, high-grade products without much fear of detection by the analytical methods at present available. Apparently the only satisfactory procedure at our disposal for securing the proper quality of geranium oil is the use of well-trained olfactories.

III.—PHENACETIN: METHODS OF ANALYSIS AND COMMERCIAL STATUS.

HISTORICAL REVIEW.

On reviewing the history of phenacetin it soon becomes apparent that, like many other useful discoveries, it was not the achievement of a single individual nor of a certain time, but was gradually evolved by the successive efforts of many minds, stimulated by the progress of correlated branches of science. It was, however, due to the patient efforts of O. Hinsberg and his associates that the production of this article on a commercial scale was made possible and its great medicinal value became known to the world.

The germ of phenacetin was sown by the celebrated French chemist, A. Cahours, during the course of a series of valuable investigations extending over a number of years. In 1843[a] appeared his "Recherches sur l'huile de Gaultheria procumbens," which work was persistently continued until 1849,[b] when he announced the discovery of phenetol ($C_6H_5OC_2H_5$, phenol ethyl ether), mononitrophenetol ($C_6H_4NO_2OC_2H_5$), and phenetidin ($C_{14}H_9NO_2C_2H_2$, old nomenclature). Two methods for preparing phenetol were given; one by heating together ethyl salicylate and baryta, and the other by simply heating barium ethyl salicylate.

Following up a suggestion of Cahours, G. Baly,[c] at the request of A. W. Hofmann, prepared phenetol by the first method described above, but called it "salithol." Baly presented his work to the London Chemical Society December 4, 1848, but it did not appear in the society's official organ until 1850. It is interesting to note in this connection that several foreign journals printed the article in 1849, giving the Quarterly Journal of the Chemical Society credit for an article that did not appear in its columns until nearly a year afterwards.

In 1851 Cahours[d] prepared phenetol by heating potassium phenolate and ethyl iodid together in a sealed tube at from 100° to 120° C. The following year appeared C. Gerhardt's[e] classic "Recherches sur les acides organique anhydres." In this investigation we find prepared for the first time acetanilid and benzanilid, by the action of acetic anhydrid and benzoic anhydrid, respectively, on anilin. This same worker[a] had made benzanilid seven years before by the interaction of benzoyl chlorid and anilin.

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[a] Comp. rend., 1843, **16**: 853; J. prakt. Chem., **29**: 197.

[b] Ann. Chim. Phys., 1849 (3), **27**: 439; J. prakt. Chem., 1850, **49**: 262; Ann. (Liebig), **74**: 314.

[c] Quart. J. Chem. Soc., 1850, **2**: 28; J. prakt. Chem., 1849, **47**: 419.

[d] Comp. rend., 1851, **32**: 60; Ann. (Liebig), **78**: 225.

[e] Comp. rend., 1852, **34**: 755; J. prakt. Chem., **56**: 321.

In 1854 J. Fritzsche began to study the action of nitric acid on phenol. His first results were presented to the St. Petersburg Academy of Sciences November 6, 1857.[b] In this communication he reported the preparation of ortho-nitrophenetol by the action of ethyl-iodid on a silver salt of ortho-nitrophenol. The ester was extracted and purified by treating the mixture with ether, evaporating the solvent, and distilling the resulting reddish-yellow fluid. The product thus obtained was nearly odorless, almost insoluble in water, but readily soluble in alcohol and ether. Boiling caustic potash solution decomposed it with difficulty.

The following year[c] the same worker discovered and described isonitrophenic acid (para-nitrophenol) and some of its derivatives. The derivative of particular interest in this connection is its ethyl ester, para-nitrophenetol, prepared as was the ortho-nitrophenetol above. Para-nitrophenetol he described as a colorless crystalline body possessing an agreeable aromatic odor, nearly insoluble in water, soluble in ether and alcohol, and melting at 57° to 58° C.

Nothing of importance appeared again until A. Groll[d] announced the preparation of ortho-amidophenetol from ortho-nitrophenetol. The latter was prepared by dissolving potassium nitrophenolate and ethyl bromid in from 3 to 4 parts of alcohol and heating the mixture for a number of hours in a sealed tube at from 140° to 160° C. Ortho-amidophenetol was made by treating ortho-nitrophenetol with metallic tin and hydrochloric acid. From this time forward the nitrophenetols were well known to organic chemists, being frequently employed by them in their research work, and probably in the industrial world. Para-nitrophenetol was used by R. Schmitt and R. Möhlau[e] in their investigation on azoxy-azo- and hydrazo-phenetol, by Schmitt[f] during his study of the constitution of the dichlorazophenols, and R. Möhlau in his dissertation[g] very fully describes ortho-nitrophenetol. The latter, in his study of ortho-diamidodiphenetol,[h] fully sets forth that the phenetols were common property.

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[a] Comp. rend., 1845, **20**: 1031.

[b] St. Petersb. Bull. Classe phys. math., 1858, **16**: 161; J. prakt. Chem., **73**: 293; Chem. Centralb. (2), **3**: 171.

[c] St. Petersb. Bull. Classe phys. math., 1859, **17**: 145; J. prakt. Chem., **75**: 257; Ann. (Liebig), **110**: 155; Jahresb. (Liebig and Kopp), **11**: 407.

[d] J. prakt. Chem., 1875 (2), **12**: 207.

[e] J. prakt. Chem., 1878 (2), **18**: 198.

[f] J. prakt. Chem., 1879 (2), **19**: 312.

[g] Dissertation, Freiburg, 1879, i, B. 27, ff, through J. prakt. Chem., 1880 (2), **21**: 318.

[h] J. prakt. Chem., 1879 (2), **19**: 381.

By allowing fuming nitric acid to act on phenetol, Cahours[a] obtained a solid and a liquid body; the former he called dinitrophenetol and the latter mononitrophenetol. E. J. Hallock[b] repeated these experiments and obtained two similar bodies. He says:

> The solid, when purified by repeated recrystallizations, both from acid and from alcohol, was proved by an ultimate analysis to be a mononitrophenetol. Its melting point, 58° C., and other physical properties coincide with that of paramononitrophenetol, prepared by Fritzsche in 1858 by the action of iodid of ethyl upon the silver salt of para-nitrophenol.

H. Andreae[c] in his excellent communication of nitro-ortho- and nitro-para-azophenetol shows that he was well acquainted with both the para- and the ortho-nitrophenetol. C. Willgerodt[d] gave a new method for preparing para-nitrophenetol, as indicated by the following equation: $C_6H_4(NO_2)Cl+C_2H_5OH=C_6H_4(NO_2)OC_2H_5+HCl$. H. Kolbe[e] gave additional methods for preparing these products. Other useful papers published bearing on these products were contributed by J. Berlinerblau,[f] C. Liebermann and St. Kostanecki,[g] and C. Willgerodt and M. Ferko.[h]

The next step involved in the production of the phenacetins is the conversion of the nitrophenetols into the amidophenetols. This is usually done by the well-known nascent hydrogen process.

Ortho-amidophenetol was first given to the world by A. Groll[i] in 1875. The same compound was produced by M. Förster[j] in 1880 and by J. Berlinerblau[f] in 1884. Meta-amidophenetol was well known to Berlinerblau,[f] and Ph. Wagner,[k] and undoubtedly to other organic chemists in the early eighties. The discovery of para-amidophenetol is generally credited to the American chemist, E. J. Hallock,[b] but R. Schmitt's[l] work on the constitution of the dichlorazophenols shows that chemists were well acquainted with this compound before the publication of Hallock's article. It was well known to Liebermann and Kostanecki[g] in 1884.

The process described in the phenacetin patent for the manufacture of para-phenetidin was old and well known prior to the date when the patent was granted—1889. Substantially the same method was described in 1884 by H. Köhler.[m]

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[a] Ann. Chim. Phys., 1849 (3), **27**: 465.

[b] Am. Chem. J., 1879, **1**: 271.

[c] J. prakt. Chem., 1880 (2), **21**: 318.

[d] Ber. d. chem. Ges., 1881, **14**: 2636; ibid., 1882, **15**: 1002.

[e] J. prakt. Chem., 1883 (2), **27**: 424; ibid. (2), **28**: 62.

[f] J. prakt. Chem., 1884 (2), **30**: 97.

[g] Ber. d. chem. Ges., 1884, **17**: 876.

[h] J. prakt. Chem., 1886 (2), **33**: 152.

[i] J. prakt. Chem., 1875 (2), **12**: 207.

[j] J. prakt. Chem., 1880 (2), **21**: 341.

[k] J. prakt. Chem., 1885 (2), **32**: 70.

[l] J. prakt. Chem., 1879 (2), **19**: 312.

[m] J. prakt. Chem., 1884 (2), **29**: 257.

Meta-acetphenetidin, or the meta variety of phenacetin, was prepared by Ph. Wagner[a] in 1885. This he accomplished by gently heating together two parts of meta-phenetidin and one part of acetic anhydrid, cooling the resulting mixture, transferring the cooled crystalline mass to a funnel, washing out part of the impurities by means of water, and finally recrystallizing from hot water. The purified, glistening crystals were white, with a tint of red, melted at 96.7° C. (uncorrected), and were difficultly soluble in water.

After having prepared para-amidophenetol, Hallock[b] experimented further with this compound and reported one observation in the following words: "This oil combines, like anilin, directly with acetyl chlorid to a crystalline solid." This crystalline solid, for the greater part, undoubtedly consisted of para-acetphenetidin, or phenacetin proper. Hallock, however, did not isolate the crystalline body and establish its physical properties and chemical composition. If he had done this it would have been a positive anticipation of the patent, and that part of the patent alleging novelty or "a new pharmaceutical product" would have had its mainstay undermined. All information about the crystalline body was left vague and inconclusive by Hallock, who evidently did not think that it possessed any value or was worth further investigation. The statement that he obtained such a body by the interaction of acetyl chlorid and para-amidophenetol was not useful to the public.

The great desideratum with the medical profession has been a safe, effective, and inexpensive antipyretic. The oldest and best febrifuge at this time (1880) was quinine sulphate, but on account of its costliness many efforts had been made by chemists either to prepare quinine artificially or to find an efficient substitute. In 1842 Gerhardt[c] distilled a mixture of quinine, water, and caustic potash and obtained a useful base which he called chinolin. Its high price from this source forbade its use medicinally, but by treating the alkaloid cinchonin in a similar manner he produced the same base. He was very much encouraged by the results of this investigation, for it was found that this comparatively cheap base and its salts were active antipyretics. Chinolin tartrate was the earliest largely used artificial febrifuge. Chinolin itself was used as a nucleus of many synthetic antipyretics or quinine substitutes. The successful preparation of synthetic quinine has been reported from time to time, but at this writing has not been accomplished. Stimulated by the success of Gerhardt, K. Hlasiwetz and L. Barth[d] distilled resins in the same manner in which quinine and cinchonin had been distilled and obtained a product which they called resorcin. This chemical was at first extolled as an antipyretic also.

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[a] J. prakt. Chem., 1885 (2), **32**: 70.

[b] Am. Chem. J., 1879, **1**: 271.

[c] Ann. (Liebig), 1842, **42**: 310; J. prakt. Chem., **28**: 65.

[d] Wien. Akad. Ber., 1864, **49**: 203; through Chem. Centralb., (2), **9**: 806.

From 1862 to 1879 there appeared to be a cessation of activities in the field of this class of remedies, but at the latter date W. Königs[a] gave a new impetus to the work by his researches on chinolin, making it synthetically on a large scale from anilin. He was soon joined in his investigations by Baeyer, Skraup, Fischer, Knorr, Körner, and others, with very fruitful results.

Doctors Fischer and Königs in their studies on the alkaloids came to the conclusion that the properties of quinine did not reside in the quinolin nucleus, but in an oxygen or hydrogen-bearing element contained in or introduced into the nucleus. With this in mind their researches were prosecuted, and a number of new bodies were discovered, only two of which, however, appeared to be successful as medicinal agents. These were oxyhydromethylchinolin hydrochlorid, prepared by O. Fischer[b] and called "Kairin," and tetrahydromethylquinolin or tetrahydroethylquinolin, made by L. Hoffmann and W. Königs[c] and named "Kairolin." The former was patented, highly extolled, and extensively advertised, and seems to have been the first medicinal chemical that was stimulated by the mercantile influence of letters patent.

In 1884, L. Knorr[d] succeeded in preparing another very efficient antipyretic and named it antipyrin. It was well covered by patents. In France, however, a patent was granted only on a process for manufacturing it as an anilin product. But antipyrin did not possess any industrial value and could not be sold as a patented remedy, for France, in the endeavor to hold the interests of suffering humanity above the interest of the individual inventor, does not grant patents on medicinal agents. The result was that antipyrin was not employed openly in France during the life of the patent.

Acetanilid was discovered in 1852 by C. Gerhardt,[e] but its antipyretic properties were not revealed until 1886, by G. Krieger.[f] It is well known by its trade name Antifebrin. This compound unfortunately is prone to induce collapse when frequently administered in large doses. It was shown twenty years previous to this time by C. D. Schroff,[g] A. Crum-Brown, and T. R. Fraser,[h] and more recently by Stolnikow,[a] that a modification of the chemical constitution of a chemical compound materially changes its physiological action. For example, the introduction of a methoxy group into morphine, converting it into codeine, materially diminishes its narcotic action.

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[a] Ber. d. chem. Ges., 1879, **12**: 453.

[b] Ber. d. chem. Ges., 1883, **16**: 712; Arch. d. Pharm. (3), **21**: 617.

[c] Ber. d. chem. Ges., 1883, **16**: 727.

[d] Ber. d. chem. Ges., 1884, **17**: 2032; J. Soc. Chem. Ind., 1885, **4**: 59.

[e] Comp. rend., 1852, **34**: 755.

[f] Centralb. f. Klin. Med., 1886, **7**: 761.

[g] Wochenblatt d. K. K. Ges. Aerzte, Wien, 1866, **6**: 157.

[h] Trans. Royal Soc. Edin., 1867 to 1869, **25**: 151 and 693; J. Anatomy and Physiol., (2), **3**: 478.

Salol was prepared by M. Nencki[b] and investigated therapeutically by Dr. Sahli.[c] This chemical was fully covered by patents.

In 1887 phenacetin, as an antipyretic, first made its public appearance through communications by G. Kobler[d] and E. Ghillany.[e] This chemical was prepared by Hinsberg. No unfavorable after symptoms were noticed in 50 cases that were treated. O. Hinsberg and A. Kast[f] reported that para-acetphenetidin (phenacetin) in doses of 3 grams acted like a strong poison, but in doses from 0.2 to 0.5 gram, exhibited to feverish persons, it was an effective antipyretic.

E. Utescher[g] reported phenacetin as a very reliable antipyretic and singularly free from secondary effects. He described it as an odorless, tasteless, white (pinkish tint), crystalline body, having a melting point of 132.5° C. When heated with sulphuric acid, ethyl acetate is produced. On heating a mixture of phenacetin, potassium hydroxid solution, and chloroform the isonitril odor is developed. Phenacetin heated with a potassium hydroxid solution liberates ethyl alcohol, which, on the addition of iodin, gives iodoform. This phenacetin was manufactured by F. Bayer & Co.

The above historical review quite fully sets forth the state of the art and knowledge prior to the application for the phenacetin patent, June 29, 1888. The various methods of making acetanilid and its physiological action were well known. Acetanilid produced unfavorable after results when given in repeated large doses. Some undesirable physiological actions had been removed or modified or mitigated by the introduction of certain well-known groups. Phenacetin has the same chemical constitution as acetanilid, excepting that one of the atoms of hydrogen of acetanilid has been substituted by an ethoxy group.

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[a] Ztschr. f. Physiol. Chem., 1884, 8: 235.

[b] Polytech. Notizbl., 1886, **41**: 176; through Chem. Centralb., (3), **17**: 751.

[c] Therap. Monatsh. Berl., 1887, **1**: 333.

[d] Ztschr. Österr. Apoth. Ver., 1887, **25**: 323.

[e] Ztschr. Österr. Apoth. Ver., 1887, **25**: 339; J. Soc. Chem. Ind., **6**: 676.

[f] Centralb. Med. Wissensch., 1887, **25**: 145.

[g] Apoth. Ztg., 1887, **2**: 436; through J. Chem. Soc. Ind., **7**: 227.

PHENACETIN PATENTS AND TRADE-MARKS.

UNITED STATES PATENT OFFICE.

Oskar Hinsberg, of Barmen, Assignor to the Farbenfabriken, Vormals Fr. Bayer & Co., of Elberfeld, Germany.

PHENACETINE.

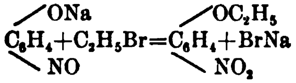
Specification forming part of letters patent No. 400086, dated March 26, 1889. Application filed June 29, 1888. Serial No. 278593. (*Specimens*)

To all whom it may concern:

Be it known that I, Oskar Hinsberg, a citizen of the Empire of Germany, residing at Barmen, in the said Empire, have invented a Useful Improvement in the Manufacture of a New Pharmaceutical Product, of which the following is a specification.

My invention relates to the production of a new pharmaceutical product, a new antipyretic and antineuralgic, obtained by reducing nitrophenetole and melting the phenetidinchlorhydrate thus formed with dried sodium acetate and acetic acid.

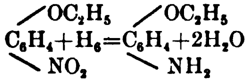
In carrying out my process practically I proceed as follows: Fifty kilos of the potassium salt of paranitrophenole are mixed with three hundred kilos of alcohol, adding forty kilos of bromaethyl. The mixture is heated in an autoclave at a pressure of three to four atmospheres during about eight hours. At this time the reaction is finished, whereby paranitrophenetole is obtained according to the following equations:



$$C_6H_4\begin{array}{l}\diagup ONa\\ \diagdown NO\end{array}+C_2H_5Br=C_6H_4\begin{array}{l}\diagup OC_2H_5\\ \diagdown NO_2\end{array}+BrNa$$

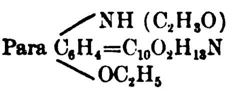
In order to separate the mononitrophenole, which has not taken any part in the process, from the ether recently formed, the solution is treated with steam. By this operation the ether distills, leaving behind the paramononitrophenole.

For the reduction of the paranitrophenetole forty kilos of this ether are mixed with sixty kilos of muriatic acid and sixty kilos of water. To this mixture are gradually added, at a temperature of 70° centigrade, twenty-five kilos of iron filings, the whole being stirred continually. As soon as the ether is entirely reduced, para-amidophenetole is obtained, as explained by the following equation:



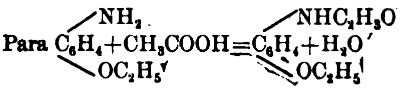
$$C_6H_4\begin{array}{l}\diagup OC_2H_5\\ \diagdown NO_2\end{array}+H_6=C_6H_4\begin{array}{l}\diagup OC_2H_5\\ \diagdown NH_2\end{array}+2H_2O$$

The solution obtained in this manner is saturated with chalk diluted with water, and for the purification of the amido compound treated with steam the distillate is absorbed in water acidulated by muriatic acid. The muriatic salt of the para-amidophenetole crystallizes in white leaves. Fifty kilos of this product are melted with one molecule of melted acetate of sodium and twenty four kilos of glacial acetic acid. The melted mass is repeatedly boiled with water and the new monoacetylparamidophenetole obtained from the filterates after cooling. It has the following chemical formula:



$$\text{Para } C_6H_4\begin{array}{l}\diagup NH\ (C_2H_3O)\\ \diagdown OC_2H_5\end{array}=C_{10}O_2H_{13}N$$

and is obtained according to the following equations:



$$\text{Para } C_6H_4\begin{array}{l}\diagup NH_2\\ \diagdown OC_2H_5\end{array}+CH_3COOH=C_6H_4\begin{array}{l}\diagup NHC_2H_3O\\ \diagdown OC_2H_5\end{array}+H_2O$$

The monoacetylparamidophenetole crystallizes in white leaves, melting at 133° to 136° centigrade. It is tasteless, little soluble in cold water, more so in hot water, but easily in alcohol, chloroform, benzole, etc. It is altogether different from the body described in the *Year Book of Pharmacy*, 1883, page 146, denominated "phenacetëine". The formula of phenacetëine is $C_{10}H_{12}O$, that of phenacetine $C_{10}H_{13}O_2N$, my product containing nitrogen contrary to phenacetëine. The phenacetëine represents a coloring matter, an armorphous carmine red powder, the acid solution of which is yellow, the alkaline raspberry red, while my phenacetine is colorless, crystallizing in white leaves, not changing color by addition of acids or alkalies.

Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is—

The product herein described, which has the following characteristics: it crystallizes in white leaves, melting at 135° centigrade; not coloring on addition of acids or alkalies; is little soluble in cold water, more so in hot water; easily soluble in alcohol, ether, chloroform, or benzole; is without taste and has the general composition $C_{10}H_{13}O_2N$.

O. Hinsberg.

Witnesses:
Wm. Diestel,
O. J. Heimpel.

A patent on phenacetin was granted to O. Klimmek,[a] of Chicago, Ill., under the chemical name oxyethylacetanilid. This patent was subsequently found to be invalid.

On reading the phenacetin patent (Hinsberg) it will be observed that this patent is for the product and the descriptive portion or specification sets forth a process by which this product is made. In an attack on the validity of the patent the patentees took shelter behind this claim, alleging that the invention resided in the product and not in the process. The patentee undoubtedly felt that there was nothing new in the process described, and it was a necessity to draft the patent in such a manner as to permit the above construction. There is nothing new in the process. Every step was well known to chemists long prior to the time of application for the patent. The only useful improvement in the process was its application on a commercial scale, and there could have been little hope of successfully defending such a process in case of an attack. The claim for the product seems to be quite safe, for there is nothing available in ordinary chemical literature that conclusively anticipated the patent. The "crystalline solid" of Hallock, spoken of above, was undoubtedly impure phenacetin, but his information concerning the product was not sufficient anticipation, in the opinion of the courts, to invalidate a useful patent.

The patent has been declared good and valid in law by the United States circuit court[b] for the eastern district of Pennsylvania, and the United States circuit court of appeals[c] for the third circuit affirmed the decision of the lower court. O. Hinsberg must therefore at present be considered the acknowledged discoverer of phenacetin.

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[a] U. S. Pat. No. 606288, June 28, 1898.

[b] Dickerson et al. *v.* Mauer, Fed. Rept., 1901, **108**: 233.

[c] Mauer *v.* Dickerson et al., Fed. Rept., 1902, **118**: 870.

Phenacetin is an eminently efficient medicinal remedy, and is consequently largely used in all parts of the civilized world. It is open to competition in every country except the United States. This situation has caused the pharmaceutical profession of the United States to chafe considerably for many years. They think such a discrimination is an injustice. In many cases the burden became unbearable, and an illegitimate product began to be smuggled into this country from the Canadian borders. At present it is mostly brought in by unauthorized agents through the customs, duty paid, and delivered to the trade at about half the price charged by the holders of the patent. Much of this smuggled phenacetin bears the same label as the authorized product, but it is expressly stated on the carton that "The resale and importation to the United States of America is prohibited." It is sometimes called "peddled" phenacetin. In some cases the dealers in these goods are not satisfied with their profits, but mix the phenacetin with acetanilid and put it up in spurious packages closely resembling the original cartons. Adulterated phenacetin has caused the druggists of this country much trouble.

For a time it was thought that phenacetin could be handled in this country under its chemical name, para-acetphenetidin, as were dermatol (bismuth subgallate), antifebrin (acetanilid), and antipyrin (dimethyloxychinizin), and as aristol (dithymol di-iodid) is at present; but the court held that the phenacetin patent covered this name[a] also. Whatever may be the status quo, it is quite evident that very little para-acetphenetidin as such finds its way into the United States except such as is brought in surreptitiously.

The words "Phenacetin" and "Phenacetin-Bayer" are protected by United States registered trade-marks, numbers 18637 and 16392, respectively. If the usual custom should prevail, the owners of these trade-marks would have the exclusive right to use these words for thirty years from date of registration, but according to certain recent United States Supreme Court decisions[b] the word "Phenacetin" becomes public property at the expiration of the phenacetin patent; otherwise it would be necessary to use the chemical name or coin a suitable one for common use, as was the case with vaseline, known in the United States Pharmacopœia, 1890, as petrolatum. The following interesting extract is taken from the Singer decision:

> TRADE-MARK—DOCTRINE AS TO USE OF NAME GIVEN TO PATENTED ARTICLE AFTER EXPIRATION OF PATENTS.—It is the universal American, English, and French doctrine that where, during the life of a monopoly created by a patent, a name, whether it be arbitrary or be that of the inventor, has become by his consent, either express or

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[a] Dickerson *v.* Tinling, Fed. Rept., 1897, **84**: 192.

[b] Official Gazette, 1896, **75**: 1703, Singer Manufacturing Company *v.* June Manufacturing Company; Official Gazette, 1901, **97**: 958, The Holzapfel's Composition Company (Limited) *v.* The Rahtjen's American Composition Company.

tacit, the identifying and generic name of the thing patented, this name passes to the public with the cessation of the monopoly which the patent created.

SAME, SAME—USE OF NAME TO DECEIVE.—Where another avails himself of this public dedication to make the machine and use the generic designation, he can do so in full forms, with the fullest liberty, by affixing such name to the machines, by referring to it in advertisements, and by any other means, subject, however, to the condition that the name must be so used as not to deprive others of their rights or to deceive the public, and, therefore, that the name must be accompanied with such identifications that the thing manufactured is the work of the one making it as will unmistakably inform the public of that fact.

On the principles embodied in the above decision the trade-marked names Antipyrin, Castoria, and Lanolin have become public property. Any one is at liberty to manufacture the above articles, with the restriction that the public must not be deceived as to the maker.

The underlying principle of our patent laws is to stimulate and protect invention. As a patent covering a new product prevents the production of the article during its life by other processes, even those which may be more economical, the inventor of a new process for the preparation of a patented product will be prevented by the owner of the product patent from practicing his new process during the life of the patent. To this extent the patent laws may be said to temporarily discourage the invention of new and more economical processes for the production of such patented products; but if to remedy such difficulty the protection by patent should be removed from this class of products these would be left unprotected as a class of useful inventions. It is generally admitted that our patent laws do work hardships in a few cases like phenacetin, but if they were modified so as to remedy such difficulties much greater ones would become involved. Though inequalities exist, our patent laws as a whole are considered by those who have made a study of the subject to be the best in the world.

The commission appointed by President McKinley to revise the United States patent and trade-mark laws did not see its way clear to make the changes desired by the American Pharmaceutical Association. For this the commission has been criticised harshly by the pharmaceutical press, which expresses surprise that the recommendations of the association named were ignored. While the recommendations appear to be reasonable, it must be remembered that our patent laws are general, and of the large number of patents granted in the United States chemicals and medicinal remedies constitute only a small percentage. Such changes would involve special (class) legislation for a comparatively few articles, which Congress is very much disinclined to enact, for experience has shown that the results are frequently far from satisfactory.

METHODS OF MANUFACTURE.

The method described in the patent does not need to be considered further. The following equations serve to illustrate the successive steps and the principles involved in the manufacture of phenacetin:

$$C_6H_4NO_2OH + 6H = C_6H_4NH_2OH + 2H_2O.$$

$$\text{p-nitrophenol} + \text{nascent hydrogen} = \text{p-amidophenol} + \text{water.}$$

$$C_6H_4NH_2OH + C_2H_5Br = C_6H_4NH_2OC_2H_5 + HBr.$$

$$\text{p-amidophenol} + \text{ethyl bromide} = \left\{\begin{matrix}\text{p-amidophenetol}\\ \text{or}\\ \text{p-phenetidin}\end{matrix}\right\} + \text{hydrobromic acid.}$$

$$C_6H_4NH_2OC_2H_5 + CH_3COOH = C_6H_4NHC_2H_3OOC_2H_5 + H_2O.$$

$$\text{p-phenetidin} + \text{glacial acetic acid} = \text{phenacetin} + \text{water.}$$

Acetic anhydrid or acetyl chlorid can be used in place of glacial acetic acid. Phenacetin is obtained pure by recrystallization from hot water. In 1888 J. D. Riedel[a] took out a German patent for the manufacture of p-amidophenetol, which is the direct antecedent of phenacetin, by reducing 10 kg of diethyldioxyazobenzol with 6 kg of tin and 50 kg of 20 per cent hydrochloric acid. As soon as the diethyldioxyazobenzol is dissolved the mixture is rendered alkaline and submitted to distillation. Para-amidophenetol is carried over with the aqueous distillate. The patentee states that this chemical is serviceable in preparing phenacetin. The reaction is represented by the following equation:

$$C_2H_5OC_6H_4NNC_6H_4OC_2H_5 + 4H = 2C_2H_5OC_6H_4NH_2.$$

According to another method,[b] 50 grams of chrysophenin, 100 grams of zinc dust, and 250 cc of hot water are mixed in a 2-liter flask and heated on a water bath with frequent agitation for one hour, then submitted to distillation. The p-amidophenetol is removed from the distillate by means of ether and the ethereal solution rendered acid with 30 cc of dilute hydrochloric acid, which converts the phenetol into an ether insoluble hydrochlorid. The ether is drawn off and the aqueous solution concentrate to crystallization.

H. N. Morse[c] in 1878 treated p-amidophenol with acetic acid with the expectation of getting an acetic acid salt of p-amidophenol, but obtained p-acetylamidophenol. In 1894 E. Taüber[d] secured letters patent in Germany for a process that converts this chemical into phenacetin. The method is as follows: Mix 150 grams of p-acetamidophenol, 165 grams of potassium ethyl sulphate, 40 grams of sodium hydrate (dissolved in 500 cc of 60 per cent alcohol) in an autoclave and heat the mixture for four hours at 150° C. On diluting the resulting solution with three parts of water the phenacetin separates out in fairly pure crystals. This appears to be the method now used in the manufacture of phenacetin.

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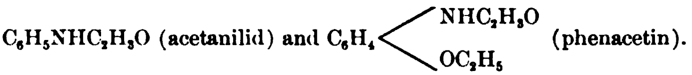
[a] D. R. Patent No. 48543, Dec. 28, 1888.

[b] Bender und Erdmann, Organische Präparate, 1894, 2: 466.

[c] Ber. d. chem. Ges., 1878, 11: 232.

[d] D. R. Patent No. 85988, June 19, 1894.

The chemical constitution of phenacetin can readily be made out from what has been said above. Its chemical names are p-acetaminophenol ethyl ether, p-acetphenetidin, p-acetamidophenetol, ethoxyacetanilid, and oxyethylacetanilid. The last two names show their close relation to acetanilid. This is also shown by the following formulæ:



$$C_6H_5NHC_2H_3O \text{ (acetanilid) and } C_6H_4{\begin{matrix} \diagup NHC_2H_3O \\ \diagdown OC_2H_5 \end{matrix}} \text{ (phenacetin).}$$

These two compounds resemble each other not only in chemical constitution, but also in their physical properties.

PHYSICAL AND CHEMICAL TESTS.

THE MELTING POINT.

Many and varied have been the tests proposed for establishing the purity of phenacetin, but at present the most reliable is its melting point, which is near 135° C. This important constant is disturbed by the presence of any impurity, such as unconverted p-phenetidin, acetanilid, antipyrin, salicylic acid, quinine sulphate, starch, milk sugar, etc. All powders so far examined in this laboratory having a melting point within 1 degree of normal have deported themselves in every respect like pure phenacetin.

The presence of unconverted p-phenetidin[a] in phenacetin is detected by melting 2.5 grams of pure crystal chloral hydrate in a test tube on a water bath, then adding 0.5 gram of phenacetin, and continuing the heating for a short time. If the phenacetin is pure, only a pinkish color develops on prolonged heating, whereas the presence of p-phenetidin (p-amido-phenetol) gives within five minutes an intensely red or reddish-violet coloration.

The one adulterant of phenacetin which appears to be difficult of detection is acetanilid. Its influence on the melting point of phenacetin was noted by E. Utescher,[b] who found that phenacetin mixed with 5 per cent of acetanilid shows, at from 113° to 114° C., minute drops of liquid on the sides of the capillary tube and melts completely at from 127° to 128° C. H. Schweitzer[c] studied the melting points of various mixtures of phenacetin and acetanilid and observed the strange phenomenon that all mixtures of these two chemicals began to melt at 92° C. Neither phenacetin nor acetanilid shows the least change at this temperature. Schweitzer thinks this test is positive in identifying mixtures of these chemicals and that further examination is useless. The following table is taken from his communication:

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[a] Pharm. Ztg., 1891, **36**: 185, through J. Soc. Chem. Ind., **10**: 799.

[b] Apoth. Ztg., 1888, **3**: 483, through Ztschr. anal. Chem., **27**: 668.

[c] J. Soc. Chem. Ind., 1895, **14**: 852.

Melting points of mixtures of phenacetin and acetanilid (Schweitzer).

No.	Mixture consisting of—		Begins to melt at—	Completely transparent at—
	Phenacetin.	Acetanilid.		
	Per cent.	*Per cent.*	°*C.*	°*C.*
1	99	1	92	134
2	95	5	92	133
3	85	15	92	128
4	66⅔	33⅓	92	126
5	60	40	92	125
6	50	50	92	122
7	40	60	92	118
8	33⅓	66⅔	92	114
9	15	85	92	110
10	5	95	92	106
11	100	0	133	134.5
12	0	100	112	114

Examinations made in this laboratory of mixtures of phenacetin and acetanilid gave the following results:

Melting points of mixtures of phenacetin and acetanilid (drug laboratory).

No.	Percentages in mixture of—		Melting point.	
	Phenacetin.	Acetanilid.	Begins to melt at—	Completely transparent at—
	Per cent.	*Per cent.*	°*C.*	°*C.*
1	90	10	96	127
2	75	25	92	121
3	50	50	90	92
4	25	75	90	99
5	10	90	95	110
6	100	0	133.5	134.8
7	0	100	112	113.8

The above observations were made several times on finely powdered anhydrous thoroughly mixed material with a standard thermometer, the entire mercurial column being in the heating medium. The melting points were taken in a long capillary tube attached to the thermometer, and both were placed in a test tube 20 cm long and 2 cm in diameter. The test tube was then filled with glycerin and placed in a liter Erlenmeyer flask nearly filled with glycerin. The temperature was gradually raised and kept uniform throughout the mass by a continual current of air. The results are not in perfect accord with Schweitzer's. The various mixtures began to soften at about 92° C., but not uniformly at that temperature.

Since these determinations were made an article by G. M. Beringer[a] has appeared, from which the following table on the melting points of mixtures of acetanilid and phenacetin is taken:

Melting points of mixtures of acetanilid and phenacetin (*Beringer*).

Acetanilid.	Shrinks.	Softens.	Melts.	Complete fusion.
Per cent.	°*C.*	°*C.*	°*C.*	°*C.*
1	126	128	131-132	134
2	115	125	130-131	132
3	110	124	128-129	131
4	105	120	125-126	130
5	100	108	112-114	124
10	100	105	110-112	120
15	95	105	110-112	120
20	92	100	105-106	118
25	90	94	95-96	115
30	90	92	94-95	115
35	85	90	92-94	108
40	84	90	91-93	106
45	82	90	92-93	100
50	80	86	90-92	96
55	77	86	90-92	95
60	82	88	90-92	94
65	82	87	90-92	94
70	87	90	91-92	95
75	88	90	92-93	96
80	88	90	91-92	99
85	90	92	95-96	105
90	92	95	100-101	108
95	96	102	107-108	111

These results do not add anything to what has already been noted, but they do show clearly, first, that chemists have not yet thoroughly learned how to determine the point at which a substance melts or begins to soften; and, second, that all mixtures of phenacetin and acetanilid do not begin to soften uniformly at 92° C.

SOLUBILITY.

The solubility of phenacetin is of service in detecting such adulterants as sodium bicarbonate, sugar, starch, quinine sulphate, and antipyrin.

CHEMICAL TESTS FOR ACETANILID IN PHENACETIN.

Numerous chemical tests have been proposed to detect the presence of acetanilid in phenacetin, but so far the ideal method has not been found. These two chemical bodies bear such a close resemblance to each other that it is difficult to detect the cheaper acetanilid in the more expensive phenacetin. The reverse is quite easy to accomplish. The following tests are given in the order of their usefulness.

BROMIN TEST (E. HIRSCHSOHN).[b]

Boil 0.1 gram of the suspected mixture with 10 cc of water for several minutes, cool, filter, and to the filtrate add bromin water until a yellow coloration is produced. With phenacetin no turbidity results, but the presence of acetanilid causes a precipitate of p-bromacetanilid which melts at from 166° to 168° C. This test is recognized in the British and German pharmacopœias.

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[a] Drug. Circular, 1903, **47**: 184.

[b] Pharm. Ztschr. f. Russ., 1888, **27**: 794.

SAPONIFICATION TEST (P. N. RAIKOW AND P. SCHTARBANOW).[a]

This test is based on the fact that acetanilid decomposes more readily with a fixed alkaline solution than phenacetin. It is applied as follows:

In a test tube of suitable size, provided with a perforated rubber stopple carrying a bent tube about 20 cm long, place 1 gram of acetanilid or phenacetin or a mixture of the two and 8 cc of a 25 per cent solution of potassium hydroxid and submit the mixture to distillation. Receive the distillate in a second test tube containing about 5 cc of a good bleaching-powder solution. With acetanilid alone a violet blue or purplish coloration develops, due to the anilin distilled; with phenacetin a brick-red opalescent solution forms by the interaction of the phenetidin and calcium hyperchlorite; with a mixture of phenacetin and acetanilid the first few drops of the distillate give the violet-blue coloration, while the latter portion of the distillate, received in a separate test tube, gives a brick-red turbidity and some coloration. With a considerable quantity of phenacetin the color becomes intensely red and the turbidity increases. In order to clearly differentiate between these two colorations it is necessary to collect the first few drops of the distillate for the acetanilid test and the latter portion for the phenacetin test. If this precaution is not carefully observed the phenetidin color reaction will obscure the anilin test, and the results are valueless. If acetanilid is not present the first few drops of the distillate will not produce any coloration with the calcium hyperchlorite solution.

G. M. Beringer[b] has studied this method very thoroughly and suggests several changes which he thinks materially improve it. By replacing the chlorinated-lime solution with a chlorinated-soda solution the end color reactions obtained become much more characteristic. With this reagent, acetanilid gives a decidedly purple tint not in the least obscured by a cloudy brownish-red, as is the case with the chlorinated-lime solution. With phenacetin the sodium-hyperchlorite solution changes to a "bright orange" (brick-red) and remains clear. The chlorinated-soda solution is considered to be a far more delicate and satisfactory reagent. Beringer says that he did not have any difficulty in detecting 3 per cent of acetanilid in phenacetin.

Another modification of this test suggested by the same writer is to heat together for one minute 0.1 gram of the substance and 3 cc of a sodium-hydroxid solution, cool thoroughly, add 5 cc of a chlorinated-soda solution, shake well, and set aside for a few minutes. If phenacetin only is present the upper layer is never of a deeper tint than yellow, but the presence of acetanilid imparts to it a purplish-red shade.

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[a] Oesterr. Chem. Ztg., 1900, 3: 125.

[b] Drug Circular, 1903, 47: 184.

Other modifications are given, but the following is considered the most delicate: Intimately mix 1 gram of sodium peroxid with 0.1 gram of the substance to be tested, place the mixture in a 15 cm test tube, and add 3 cc of water. After the vigorous action ceases, shake thoroughly, cool, and add 5 cc of sodium-hyperchlorite solution, again shake vigorously, and set aside. If the phenacetin is pure the liquid remains colorless, or, at most, after long standing assumes a pale-yellow color, while the presence of acetanilid gives a purplish-red tint shading to pink, depending upon the amount of the adulterant present.

Judging from the chemical composition of phenacetin and acetanilid and the final color reactions, it is quite probable that the sodium peroxid acts mainly as a saponifying agent, as does potassium hydroxid in the original method.

MERCUROUS NITRATE TEST (P. C. PLUGGE).[a]

Boil together 0.5 gram of phenacetin and 8 cc of water, cool and filter. To the filtrate add a fragment of potassium nitrite and 0.5 cc of dilute nitric acid and boil the mixture a few minutes. Then add 1 cc of mercurous nitrate solution containing nitrous acid, boil again, and if a red color develops acetanilid is indicated.

IODOPHENOL TEST.[b]

This test was adopted by the German Pharmacopœial Commission for the identification of acetanilid, and is recognized by the fourth edition of the German Pharmacopœia, but the reaction has been proved to be worthless. Phenacetin responds affirmatively to this test, which is executed as follows:

Heat together for a few minutes 0.2 gram of acetanilid and 2 cc of 25 per cent hydrochloric acid. A clear solution results, which, with 4 cc of a 5 per cent solution of carbolic acid and a suitable amount of a good calcium hypochlorite solution (1 in 10) gives a dirty violet-blue color. On rendering this solution alkaline with ammonia water an indigo blue develops. The test has been modified in various ways, but so far the results are not satisfactory.

ISONITRIL REACTION.

This test was originally proposed by A. W. Hofmann[c] as characteristic of anilin. It has, however, been found that primary amines in general respond to this reaction. It is therefore a group reaction and can not with safety be employed to detect one compound containing an amido group in the presence of another compound containing the same group, even though one is more readily acted on by the agents employed than the other. The test is applied by heating together for a few minutes about 0.5 gram of the substance and 5 cc of a 10 per cent solution of sodium hydroxid, then cautiously adding a few drops of chloroform and setting the mixture aside for a few minutes. If a primary amine is present the characteristic offensive odor of phenylcarbamine can readily be detected. This reaction has received official sanction in the pharmacopœias of the United States, England, and Germany, and is so frequently cited as suitable for detecting acetanilid in phenacetin that a short résumé of some of the misleading statements made concerning it is desirable.

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[a] Arch. d. Pharm., 1890, **228**: 9; J. Anal. Appl. Chem., **7**: 77.

[b] Arch. d. Pharm., 1887, **225**: 1042.

[c] Ber. d. chem. Ges., 1870, **3**: 767.

CONFLICTING STATEMENTS REGARDING THE ISONITRIL REACTION.

As early as 1887 Utescher[a] found that after heating phenacetin, sulphuric acid, and alcohol together, rendering the mixture distinctly alkaline with a fixed alkali, and adding a few drops of chloroform, the repugnant odor of carbylamine was developed. The sulphuric acid probably decomposes the phenacetin into acetic acid and phenetidin (C_6H_4 NH_2 OC_2H_5).[b] Schwartz[c] states that a simple method for determining acetanilid in phenacetin is by means of the isonitril reaction. W. Lenz,[d] in his review of special analytical methods, says that it is self-evident that for the detection of acetanilid in phenacetin the well-known "isonitril reaction" can be employed. Again, this author states that a mixture of acetanilid and phenacetin will give the "isonitril reaction." C. Platt[e] says:

> Another test useful also in detecting small quantities of acetanilid in the presence of phenacetin is to treat the mixture with caustic soda or potash in the presence of chloroform, when, if acetanilid be present, the characteristic odor of isonitril is given off. If phenacetin be heated with alcohol and sulphuric acid the characteristic odor of ethyl acetate is observed; by heating the solution thus formed with caustic potash and chloroform, the carbylamine reaction is obtained.

In an article entitled "Adulteration and Substitution of Drugs," by Schweitzer,[f] the following statements are found:

> Acetanilid is identified * * * by heating 0.10 gram of the powder with 1 cc of NaOH (15 per cent NaOH) and three drops of chloroform, whereupon the offensive smell of phenyl-isocyanide is given off. * * * For the identification of the mixture (acetanilid and phenacetin) it is sufficient to state that the substance begins to melt at 92° C. and becomes completely transparent below 134° C. On heating 0.10 gram of powder with 1 cc of caustic soda (15 per cent NaOH) and 3 drops of chloroform the offensive smell of phenyl-isocyanide is observed.

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[a] Apoth. Ztg., 1887, **2**: 436, through J. Soc. Chem. Ind., **7**: 227.

[b] Ann. (Liebig), 1899, **309**: 233.

[c] Pharm. Ztg., 1888, **33**: 357.

[d] Ztschr. anal. Chem., **27**: 665.

[e] J. Anal. Appl. Chem., 1893, **7**: 77.

[f] J. Soc. Chem. Ind., 1895, **14**: 852.

According to G. Guasti[a] phenacetin gives the phenyl-carbylamin reaction. F. S. Hyde[b] says: "Contrary to some writers, phenacetin will give the isonitrile test, and hence can not be distinguished from acetanilid by this reaction." F. X. Moerk[c] obtained this reaction in the usual way with both acetanilid and phenacetin, but says that 1 per cent of the former is readily detected in the latter when a solution of potassium permanganate is added to destroy odors that are formed by other bodies and which interfere with the test. P. W. Squire[d] reports satisfactory results with this modification.

The isonitril reaction is recognized as an identifying test for acetanilid by the present British, German, and United States pharmacopœias. Neither of the two former pharmacopœias mentions this test in connection with phenacetin, and the latter does not recognize this chemical. Such careful workers as Helbing, Fischer, and Flückiger state that phenacetin gives the isonitril reaction.

From this array of contradictory statements it is not surprising that a worker not thoroughly familiar with the literature of this subject should make the error of basing his conclusions on this reaction. These discrepancies are undoubtedly due to the fact that phenacetin is less readily decomposed with a fixed alkali solution than is acetanilid. The conditions under which the test has been applied by the various workers have not been uniform, and indeed it would be difficult to prescribe proper limitations, as has been discovered in the drug laboratory. The test has proved itself to be unreliable for the purpose of detecting acetanilid in phenacetin.

COMMERCIAL SAMPLES.

VARIOUS LABELS.

Original 1-ounce packages of the authorized phenacetin were purchased in Washington, D. C., while the unauthorized product and p-acetphenetidin were secured in Philadelphia, Pa. This is not intended to imply that the druggists of Philadelphia were trafficking in the forbidden phenacetin while those in Washington were not.

All samples of the regular article were marked as follows:

Trade-marked name: Phenacetin. Registered trade-mark, 1 Ounce Phenacetin-Bayer. Patented March 26, 1889, U. S. Patent Nr. 400086. Manufactured by Farbenfabriken, vorm. Frieds. Bayer and Co., Elberfeld, Germany, for United States patentee. This package is sold to the owner of U. S. Patent Nr. 400086 March 26, 1889, the Farbenfabriken of Elberfeld Co., 40 Stone Str., New York City.

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[a] Selmi, 1894, **4**: 96, through J. Soc. Chem. Ind., **14**: 77.

[b] J. Amer. Chem. Soc., 1895, **17**: 933.

[c] Am. J. Pharm., 1896, **68**: 89.

[d] Squire's Companion Brit. Pharm., 1899, 17th ed., p. 6.

A general registered trade-mark, No. 31422, consisting of the figure of a winged lion resting one forepaw on a globe while the other paw grasps a caduceus, is also found on each package.

The cartons of the irregular product are plainly marked as to the manufacturer and source of production. Each package is labeled in English, French, and German as follows:

"Patented in the United States of America, Nr. 400086. Phenacetine-Bayer. The resale and importation to the United States of America are prohibited."

The sample purchased by its chemical name bore the following label:

"1 lb. Paracetphenetidine Powder B. P. This serves to remind that this article must not be sold to the United States, it being patented in that country. Made in Germany. E. Merck. Darmstadt."

ANALYTICAL RESULTS.

The tests applied to these samples were such as have been found of service in establishing the quality of phenacetin, and include physical appearance, melting point, solubility, and the presence of p-phenetidin.

Analyses of commercial samples of phenacetin.

Number on package.	Kind.	Physical appearance.	Melting point.	Solubility.	P-phenetidin.
			°C.		
1726	Authorized	Micaceous scales	134.8	Normal	None.
1743	do	do	135.0	do	Do.
1782	do	do	135.0	do	Do.
04082	Paracetphenidin	Micaceous scales (powdered)	134.9	do	Do.
155641	Unauthorized	Micaceous scales	134.6	do	Do.
339232	Authorized	do	135.0	do	Do.
339243	do	do	134.4	do	Do.
339255	do	do	134.6	do	Do.
845388	Unauthorized	Micaceous scales (some large)	[a]122-123	do	Do.

[a] Began to soften at 94° C.

All of these samples are of satisfactory quality except No. 845388, which is a mixture of phenacetin and acetanilid, containing about 20 per cent of the latter. This sample and the paracetphenidin were purchased at the same place, while the other unauthorized sample was obtained from a different source of supply. On examining the carton containing the mixture of phenacetin and acetanilid it appeared to be spurious. The two empty cartons containing the unauthorized phenacetin were sent to the patentee's agent in New York with the request that he express an opinion as to their genuineness. These packages were immediately returned, No. 845388 being marked counterfeit, the other genuine. The bogus carton resembled the genuine one very closely, though there was a difference in color, style of type, and the ornamental lines employed.

COMMERCE IN PHENACETIN.

United States letters patent grant to an inventor "the exclusive right to make, use, and vend" his "invention throughout the United States and the Territories thereof." This clause effectively restricts to the inventor, his heirs, or assignees, the right to import his patented product into the United States when manufactured abroad. According to certain decisions[a] it is held by some that if the article is purchased from the patentees, tribute has been paid to the monopoly and the right is therefore acquired to import, use, and sell the article, bought elsewhere, within the United States. This privilege, however, does not obtain when purchases are made from others than the owners of the patent, because they have not then received any remuneration for the article so purchased.[b] If these principles governed the phenacetin trade the present strained situation would not exist.

Another factor which has assumed considerable proportions in the commercial world must be considered. The patentee has the right and power to sell his product subject to the express conditions that it must not be imported into the United States or sold here. He has the same vested right to sell the product with restrictions and limitations that he has to sell it at all. It is a matter of record that the patentees of phenacetin expressly state on every package sold in foreign countries that its importation into the United States and resale there is prohibited. Manufacturers of p-acetphenetidin specify on every package that it must not be sold in the United States. The following extracts from the decisions of the United States circuit court of appeals[c] express the rulings on this point in a very succinct manner:

> 1. One purchasing in a foreign country an article protected by a United States patent, from persons other than the owner of the United States patent or his vendees, can not import and sell the same in this country without infringing the United States patent.
>
> 2. One purchasing in a foreign country, from the owner of the United States patent, patented goods having marked upon them a condition that they should not be imported into the United States, can not import and sell them here without being guilty of infringement.

Judging from past reports and recent developments, our excise laws have been grossly violated by the smuggling of phenacetin, which is clandestinely brought into the country in violation of our customs laws, or is regularly imported, duty paid, and secretly sold to consumers. Pharmaceutical journals recount the arrests of numerous vendors of this illicit product. These smugglers have been apprehended in every section of the Union, and many litigations have been instituted, numerous injunctions issued, and fines imposed.

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[a] Cited in Dickerson *v.* Tinling, Fed. Rept., 1898, **84**: 192.

[b] Dickerson *v.* Tinling, Fed. Rept., 1898, **84**: 192.

[c] Dickerson *v.* Tinling, Fed. Rept., 1897, **84**: 192.

The collector of customs of the port of New York seized and confiscated illegally imported phenacetin, advertised it for sale in the customary manner, and sold it at public auction in that city in 1898. The purchaser at the time of the sale knew that the goods were smuggled, and infringed the patent. Before he could resell his purchase the representative of the patentees served an injunction preventing such sale. The case was taken into the United States circuit court, and the defendant enjoined from directly or indirectly using or selling the patented drug phenacetin. An appeal was taken to the United States circuit court of appeals, and this court affirmed the order of the lower court.[a] The defendant claimed, first, that the passage of the phenacetin through the hands of the Federal Government in some way abrogated the rights of the patentee; and, second, that by the condemnation proceedings and statutory notice the title of the property passed to the purchaser without incumbrance or reservation.

In view of the decision given above, it seems that these contentions do not obtain in the case of patented articles of the phenacetin type. The decision further states that the patentee "has no title to or lien on, or legal or equitable interest in the infringing property." The purchaser filed an application for a refund of the buying price, but the Treasury Department reported that it could not comply with this request,[b] as the law does not provide for refunds under any circumstances. The defendant refused to comply with the order to surrender the goods purchased from the Government. This curious legal entanglement as to the disposition of smuggled patented drugs is at present receiving the consideration of the Attorney-General. The issue is of the greatest importance, and has attracted the attention not only of the Treasury officials, but of the Patent Office authorities as well.

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[a] Dickerson *v.* Sheldon, Fed. Rept., 1900, **98**: 621.

[b] Oil, Paint, and Drug Reporter, August, 1903, **64**: 24.

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U. S. DEPARTMENT OF AGRICULTURE

BUREAU OF CHEMISTRY—BULLETIN NO. 81

H. W. WILEY, Chief of Bureau

PROCEEDINGS

OF THE

TWENTIETH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

WASHINGTON, D. C., NOVEMBER 19, 20, AND 21, 1903

EDITED BY

HARVEY W. WILEY

Secretary of the Association

WASHINGTON
GOVERNMENT PRINTING OFFICE
1904

LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., March 4, 1904.

Sir: I have the honor to transmit herewith the manuscript of the proceedings of the twentieth annual meeting of the Association of Official Agricultural Chemists, with the request that it be published as Bulletin No. 81 of this Bureau.

Respectfully,

H. W. Wiley,
Chief of the Bureau of Chemistry and Secretary of the Association of Official Agricultural Chemists.

Hon. James Wilson,
Secretary of Agriculture.

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PROCEEDINGS OF THE TWENTIETH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

THURSDAY—MORNING SESSION.

The twentieth annual convention of the Association of Official Agricultural Chemists was called to order by President R. J. Davidson at 10.15 o'clock on the morning of November 19, 1903, in the lecture hall of the Columbian University.

The following members and visitors were in attendance during the sessions of the convention:

MEMBERS AND VISITORS PRESENT.

Adams, Arthur B., Treasury Department, Washington, D. C.
Allen, William P., New Jersey Agricultural Experiment Station, New Brunswick, N. J.

Bache, A. W., U. S. Department of Agriculture, Washington, D. C.
Badger, E. F., State Board of Health, Providence, R. I.
Baskerville, Charles, University of North Carolina, Chapel Hill, N. C.
Beal, W. H., U. S. Department of Agriculture, Washington, D. C.
Benjamin, Marcus, Smithsonian Institution, Washington, D. C.
Bernays, Walter, City Chemist, St. Louis, Mo.
Bigelow, Willard D., U. S. Department of Agriculture, Washington, D. C.
Blake, F. E., U. S. Department of Agriculture, Washington, D. C.
Boyce, James, American Cotton Oil Company, Chicago, Ill.
Breazeale, J. F., U. S. Department of Agriculture, Washington, D. C.
Brooke, J. C., U. S. Department of Agriculture, Washington, D. C.
Brooks, R. O., 24 Jarvis place, Trenton, N. J.

Calm, C. E., 190 Michigan street, Chicago, Ill.
Cameron, F. K., U. S. Department of Agriculture, Washington, D. C.
Carpenter, F. B., Virginia-Carolina Chemical Company, Richmond, Va.
Cavanaugh, G. W., Cornell University Experiment Station, Ithaca, N. Y.
Chace, E. M., U. S. Department of Agriculture, Washington, D. C.
Chamberlain, J. S., U. S. Department of Agriculture, Washington, D. C.
Chesnut, V. K., Agricultural Experiment Station, Bozeman, Mont.
Church, C. G., U. S. Department of Agriculture, Washington, D. C.
Coates, C. E.. State University, Baton Rouge, La.
Collins, A. T., The Colburn Company, Philadelphia, Pa.
Cook, F. C., U. S. Department of Agriculture, Washington, D. C.
Crampton, C. A., Treasury Department, Washington, D. C.
Cushman, A. S., U. S. Department of Agriculture, Washington, D. C.

Davidson, R. J., Agricultural Experiment Station, Blacksburg, Va.
Dennis, L. M., Cornell University, Ithaca, N. Y.
Donk, M. G., Assistant State Chemist, Tallahassee, Fla.
Doolittle, R. E., State Analyst, Dairy and Food Department, Lansing, Mich.
Dorset, M., U. S. Department of Agriculture, Washington, D. C.
Doyle, A. M., U. S. Department of Agriculture, Washington, D. C.
Drueding, C. F., Yocum Leather Trade Laboratory, Newark, N. J.
Dubois, W. L., U. S. Department of Agriculture, Washington, D. C.
Dyck, G. E., Official Chemist, Produce Exchange, New York, N. Y.

Emery, J. A., U. S. Department of Agriculture, Washington, D. C.
Ewell, E. E., German Kali Works, Atlanta, Ga.

Failyer, G. H., U. S. Department of Agriculture, Washington, D. C.
Fields, John, Agricultural Experiment Station, Stillwater, Okla.
Fischer, Richard, State Chemist, Madison, Wis.
Foster, A. B., Agricultural College, College Park, Md.
Frear, Wm., State College, Pennsylvania.
Friend, G. C., Union Abattoir Company, Baltimore, Md.

Gascoyne, W. J., 23 South street, Baltimore, Md.
Gibboney, James H., Agricultural Experiment Station, Blacksburg, Va.
Gore, Herbert C., U. S. Department of Agriculture, Washington, D. C.
Gough, T. R., Agricultural Experiment Station, College Park, Md.
Gray, J. P., Agricultural Experiment Station, College Park, Md.
Grindley, H. S., University of Illinois, Urbana, Ill.
Gudeman, Edward, 704 Rialto Building, Chicago, Ill.

Hand, W. F., Agricultural College, Miss.
Hardin, M. B., Agricultural Experiment Station, Clemson College, S. C.
Hartwell, B. L., Agricultural Experiment Station, Kingston, R. I.
Haskins, H. D., Hatch Experiment Station, Amherst, Mass.
Haywood, J. K., U. S. Department of Agriculture, Washington, D. C.
Herff, B. von, German Kali Works, New York, N. Y.
Hillebrand, W. F., U. S. Geological Survey, Washington, D. C.
Hiltner, R. S., Treasury Department, Washington, D. C.
Hird, J. D., Health Department, Washington, D. C.
Hollister, F. M., Agricultural Experiment Station, Burlington, Vt.
Hopkins, C. G., Agricultural Experiment Station, Urbana, Ill.
Houghton, H. W., U. S. Department of Agriculture, Washington, D. C.
Howard, B. J., U. S. Department of Agriculture, Washington, D. C.
Hurst, L. A., U. S. Department of Agriculture, Washington, D. C.
Huston, H. A., German Kali Works, St. Louis, Mo.

Jenkins, E. H., Agricultural Experiment Station, New Haven, Conn.
Jones, E. E., U. S. Department of Agriculture, Washington, D. C.

Kebler, Lyman F., U. S. Department of Agriculture, Washington, D. C.
Kerr, G. A., Acme Bark Extract Company, Damascus, Va.
Kerr, R. H., Agricultural Experiment Station, College Park, Md.
Kilgore, B. W., State Chemist, Raleigh, N. C.
Knight, C. P., U. S. Department of Agriculture, Washington, D. C.
Krug, W. H., A. Klipstein & Co., New York, N. Y.

Langworthy, C. F., U. S. Department of Agriculture, Washington, D. C.
Lawson, H. W., U. S. Department of Agriculture, Washington, D. C.
Leach. A. E., State Board of Health, Boston, Mass.

LeClerc, J. A., U. S. Department of Agriculture, Washington, D. C.
Lipman, J. G., Agricultural Experiment Station, New Brunswick, N. J.
Long, J. H., Northwestern University, Chicago, Ill.
Lord, E. C. E., U. S. Department of Agriculture, Washington, D. C.

McCarthy, E. R., Acme Bark Extract Company, Damascus, Va.
McDonnell, H. B., Agricultural Experiment Station, College Park, Md.
Magruder, E. W., Department of Agriculture, Richmond, Va.
Mason, G. F., D. J. Heinz Company, Pittsburg, Pa.
Mewborne, R. G., Kentucky Tobacco Product Company, Louisville, Ky.
Moore, C. C., U. S. Department of Agriculture, Washington, D. C.
Morse, F. W., Agricultural Experiment Station, Durham, N. H.
Mosbaugh, F. R., Huntsville and Bracebridge Tanning Company, Huntsville, Canada.
Mosser, T. J., State College, Pa.
Mudge, C. W., Agricultural Experiment Station, Geneva, N. Y.
Munson, L. S., U. S. Department of Agriculture, Washington, D. C.

Nichols, M. F., Leather Trades Laboratory, Grand Rapids, Mich.
Noyes, W. A., Bureau of Standards, Washington, D. C.

Page, L. W., U. S. Department of Agriculture, Washington, D. C.
Patrick, G. E., U. S. Department of Agriculture, Washington, D. C.
Patterson, H. J., Agricultural Experiment Station, College Park, Md.
Penny, C. L., Agricultural Experiment Station, Newark, Del.
Perkins, W. R., Agricultural Experiment Station, Agricultural College, Miss.
Price, H. L., Virginia Polytechnic Institute, Blacksburg, Va.
Price, T. M., U. S. Department of Agriculture, Washington, D. C.
Putnam, Alice, Anacostia, D. C.

Reed, H. C., Stamford Manufacturing Company, Stamford, Conn.
Richardson, Clifford, New York Testing Laboratory, Long Island City, N. Y.
Richtmann, W. O., 1615 Florida avenue, Washington, D. C.
Robb, J. B., Agricultural Experiment Station, College Park, Md.
Robison, F. W., Agricultural Experiment Station, Agricultural College, Mich.
Ross, B. B., State Chemist, Auburn, Ala.
Runyan, E. G., U. S. Inspector Gas and Meters, Washington, D. C.

Sawyer, H. E., 163 C street, South Boston, Mass.
Schreiver, Oswald, U. S. Department of Agriculture, Washington, D. C.
Scovell, M. A., Agricultural Experiment Station, Lexington, Ky.
Seidell, Atherton, U. S. Department of Agriculture, Washington, D. C.
Shutt, F. T., Central Experimental Farm, Ottawa, Canada.
Simons, F. D., Treasury Department, Washington, D. C.
Smith, P. H., Hatch Experiment Station, Amherst, Mass.
Smith, B. H., U. S. Department of Agriculture, Washington, D. C.
Smith, J. G., U. S. Department of Agriculture, Washington, D. C.
Snyder, Harry, University of Minnesota, St. Anthony Park, Minn.
Stilwell, A. J., Leather Trade Counsel, Dubois, Pa.
Steinharter, J. J., Shoe and Leather Reporter, Philadelphia, Pa.
Stokes, H. N., Bureau of Standards, Washington, D. C.
Stone, W. E., Purdue University, Lafayette, Ind.
Straughn, M. N., Agricultural Experiment Station, College Park, Md.
Street, J. P., Agricultural Experiment Station, New Brunswick, N. J.

Talbot, H. P., Massachusetts Institute of Technology, Boston, Mass.
Teas, W. H., Elk and Penn Tanning Companies, Ridgway, Pa.
Tolman, L. M., U. S. Department of Agriculture, Washington, D. C.
Trescot, T. C., U. S. Department of Agriculture, Washington, D. C.

Van Dyke, J. H., U. S. Department of Agriculture, Washington, D. C.
Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
Veitch, F. P., U. S. Department of Agriculture, Washington, D. C.
Voorhees, E. B., Agricultural Experiment Station, Washington, D. C.

Warner, H. J., U. S. Department of Agriculture, Washington, D. C.
Weber, H. A., State University, Columbus, Ohio.
Weber, F. C., U. S. Department of Agriculture, Washington, D. C.
Wharton, W. R., Agricultural College, College Park, Md.
Wheeler, H. J., Agricultural Experiment Station, Kingston, R. I.
White, H. C., Athens, Ga.
Wiley, H. W., U. S. Department of Agriculture, Washington, D. C.
Wiley, S. W., American Agricultural Chemical Company, Baltimore, Md.
Williams, C. B., Department of Agriculture, Raleigh, N. C.
Wilson, H. T., Buena Vista Extract Company, Buena Vista, Va.
Winton, A. L., Agricultural Experiment Station, New Haven, Conn.
Withers, W. A., Agricultural Experiment Station, Raleigh, N. C.
Woll, F. W., Agricultural Experiment Station, Madison, Wis.
Woods, C. D., Agricultural Experiment Station, Orono, Me.

The order of business was as follows:

ORDER OF BUSINESS.

The president's address.
Appointment of committees.
Reports of the referees, in the following order:

1. Report on nitrogen.
2. Report on potash.
3. Report on phosphoric acid.
4. Report on soils.
5. Report on ash.
6. Report on foods and feeding stuffs.
7. Report on food adulteration.
8. Report on dairy products.
9. Report on sugar.
10. Report on tannin.
11. Report on insecticides.
12. Reports of special committees (food standards, fertilizer legislation).
13. Reports of committees A, B, and C, on recommendations of referees.

PRESIDENT'S ADDRESS

GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: At the opening of this, the twentieth annual convention of our association, it will not be amiss for us to consider a few points in connection with the work which first brought this association into existence. The object of this work was to secure uniformity of methods for the determination of the valuable ingredients in commercial fertilizers. The scope of the association was broadened later, so that, as the constitution now reads, its objects are: "To secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists."

How well the first part of this work, in regard to uniformity and accuracy of methods, has been done is fully attested by our official methods, which are now universally acknowledged as the proper legal methods for the determination of the constituents of commercial fertilizers. But with the advancement of the work in this direction other problems have appeared which are now occupying the attention of our referees, such as the determination of iron and aluminum in phosphates, and of phosphoric acid in basic slag. These points will in time, I have no doubt, be properly provided for in our scheme of analysis.

It is not alone the question of chemical analysis to which I wish to call attention, but also that of the establishment of uniform laws and proper standards for the inspection of commercial fertilizers, a matter intimately connected with and dependent upon chemical work. This is a vital question in connection with our duty as official chemists, and a more troublesome one perhaps could not be brought up, since so many varied conditions must be considered.

Many of our States now have laws for the regulation of the sale of commercial fertilizers, but between the 23 whose laws I have examined there is no mutual agreement either as to requirements or form of statement of composition. When we consider the immense sums of money annually invested in these materials and the large number of people who purchase them, and how few of this number have a clear understanding of the character of fertilizing materials, it behooves this association to exert all its influence to make these laws conform to some definite standard, and to aid as far as possible in having plain, simple, and intelligible statements made in regard to the constituents contained therein. At this point it might be well to mention the fact, which is well known to most of the members of the association, that our National Government has recognized this body as the proper one to aid in the formation of standards for pure foods. It would, therefore, seem reasonable to assume that the several States interested in the question of fertilizer laws would be very glad to make use of a similar standard of requirements on this subject prepared for their use by this association. These standards have been prepared by our committee on uniform fertilizer legislation, and their report was adopted by the association five years ago. It has also been adopted by the American Association of Agricultural Colleges and Experiment Stations. Although five years have elapsed since this report was published, the State laws are still far from uniform, and some passed within the last year, although the question of uniform standards was brought up, still are framed according to the old ideas, although some of the new requirements were adopted. Why is this? It certainly is not due to the lack of importance of the subject. It is true the conditions existing in the various States are widely different; yet I do not think this is the true reason. Let us look for another. As in all the work of the association individual effort is very important—the results falling short of what they should be when it becomes weak—and in this case I think individual effort is lacking. Our committee has done all in its power to have this work brought to the attention of the authorities in those States where requests for information were made, and one or two of our members have exerted their influence with good effect. But every member of this body should use his influence in his State to have the question of the modification of existing laws, or the enactment of new ones conforming to the standards adopted by this association, brought before the proper authorities at the proper time.

I suppose most of the members are familiar with the standards that have been adopted on this subject, so I will not quote all of them; but I would like to make a comparison between some of the requirements and those found in a number of our State laws. The question of nitrogen is one of the most important; so let us consider it. Our standard requires "total nitrogen guaranteed in all cases, and nitrogen in the form of nitrates or ammonium salts guaranteed separately if the manufacturer desires credit therefor." This latter point is a very important one, and the constant complaint of the manufacturer in many of our States is that he gets no credit for nitrogen as nitrate. Of the 23 State laws examined 8 only require the per cent of available nitrogen, and that expressed as total nitrogen only. Nine prohibit the use of leather in any form, and 3 leather, wool waste, hair horn, and other inert nitrogenous material, unless a printed explicit statement is attached to each package, or such a statement made to the commissioner or board of agriculture. One prohibits the use of leather in any form, and 2 prohibit leather, hair horn, and hoof meal, etc. Eleven (about one-half) require a statement of per cent of nitrogen; 7 require per cent of nitrogen or its equivalent of ammonia; and 3 require statement of per cent as ammonia. No State law examined requires the separation of the different forms of nitrogen. As nitrogen is the most expensive constituent of commercial fertilizers, the greatest safeguards as to its condition and use should be placed about it, for the sake of both purchaser and honest manufacturer. But many of our State laws leave it so unguarded that it may be furnished in forms of practically no value for fertilizing purposes, while the purchaser has to pay as if it were of the highest value. In stating percentages only minimum amounts should be given, but of the 23 laws examined, only 6, or about one-fourth, called for the minimum alone.

I would like, therefore, to call the attention of every member of this association who is connected in any way with official fertilizer control in his own State to the necessity of attending to this important part of his work. It is the duty of the official chemist to prevent adulteration and fraud by every legitimate method available. I should like also to call attention to a very important point, and our committee might take it under consideration, which should be made a part of the standards on this subject. This is the limit of error or deficiency which should be allowed on the various constituents in commercial fertilizers and fertilizing materials. Cattle foods might be included also. There is a very much wider difference on this point in our different State laws than on any other. Of the 23 examined, 7 have vague enactments, such as "* * * shall lack materially any valuable ingredient." Such a statement is very indefinite, for who is to judge where "immaterial lack" ends and "material" begins. Five have a limit based on percentage composition, 3 on commercial valuation. Of those based on percentage composition, 2 allow a limit of 1 per cent. This is a rather liberal allowance, and unscrupulous manufacturers could make money by guaranteeing and charging for, say, 2 per cent of the nitrogen and only giving 1 per cent. One law allows one-fourth of 1 per cent on all constituents, and one allows one-half of 1 per cent on 2 constituents and one-third on the other. Of those based on commercial valuation one reads: "When it is essentially below guaranteed commercial value." Two allow a deficiency of 3 per cent below guaranteed value, and one 10 per cent below guaranteed total commercial value. No doubt it would be a great relief to all parties concerned, and especially to the chemist, to have some fixed and definite standard. Of the various methods now used for determining deficiency, the one based on percentage composition is undoubtedly the best, and would, in my opinion, be the most agreeable to all parties concerned.

The question of determining the different forms of nitrogen in fertilizers has been raised by our resolutions, and yet with one or two exceptions the chemist does not determine the amounts of nitrogen as nitrate or ammonia salts separately, but simply states his results as total nitrogen. In some cases the total is given as ammonia. The purchaser is therefore left completely ignorant of the value of the material. The following resolution was adopted by the association in 1900: "It is desirable that the form of nitrogen should be determined and reported on in analysis of fertilizers." That this has not been done certainly may be the fault of our State laws in not urging it, but I think it is also to some extent the fault of the individual chemist who is making the analysis. The law does not say that he shall not make these separate determinations, therefore, in the interest of the people, both manufacturers and consumers, he should do this for their information; and by so doing he would in time create such a demand in his State for this information that the people would have it embodied in the law. In order to have laws of any sort we must educate public sentiment and create a demand for the law before we get it, and this is true in the case of our fertilizer work. If we want uniform fertilizer laws we must see to it that our people, and especially those directly concerned, should have all the information necessary to a correct understanding of the merits of the question. This can be done more effectually by the official chemists in their various reports, bulletins, and lectures at farmers' institutes than in any other way, because the people have confidence in us, and know that our aim is for the greatest good of the greatest number, and that we have no special ax to grind.

There should also be a movement to prohibit the misnaming of fertilizers; such as the term guano for an artificial mixture of phosphoric acid, nitrogen, and potash, and the use of the term dissolved bone, for dissolved phosphate rock. I know the association has no power in this matter save that of influence, but its strength as a scientific, conscientious, and conservative body, having the best interests of the people at heart, is recognized throughout the country, and it is therefore expected to take the initiative in all lines of work with which the official chemist has to deal.

In this connection how prophetic sounds a statement made in 1889 by our lamented associate, Dr. J. A. Myers, in his address as president of the association:

> If this association continues to maintain its proper attitude and high sense of the responsibilities resting upon it, we may safely say that the time is not far distant when its deliberations will virtually become the law regulating the sale of nearly every agricultural and food product offered in our cities.

The growth of the association in amount of work and in numbers has kept pace with the importance of its mission. In 1893 there were seven reporters on the various subjects then under consideration, and these are still before the association, but the subjects have been enlarged and the number increased until now we have eleven referees and thirty-four associates, and still the work and responsibilities multiply. We are to have at this meeting reports by the various associate referees on food adulteration, a comparatively new subject undertaken by the association only a year or two ago. This is the beginning of our work toward the adoption of uniform methods of analysis and standards for food products, and, as stated before, the results of this work will be used in the execution of a national pure-food law when passed.

But with all the increased amount of work that is being done there is still a great deal more ahead of us. If we expect to live and grow we must enlarge our work and our responsibilities; we must not stand still and live on what has been done. More investigation work is needed, and each and every member of the association should feel it his duty to do something each year, either as an individual worker on some special line or in assisting the referees in their efforts to improve and add to our present methods of analysis.

In looking over the list of members who have attended our last two meetings, the eighteenth and nineteenth annual conventions, we find there were present at the former meeting 118 and at the latter 100 members. How many of these took an active part in the work of assisting our referees? The largest number working on any one subject is about twelve, and on the subjects of soil and insecticides only about four or five. This looks like lack of interest in the referees' work. Of course, it is not expected that everyone will do work for all the referees, but it is expected that everyone will do some work each year on some one or more of the subjects under consideration by the association.

Another point of interest in connection with our last two meetings is the small amount of time given to discussion. The opportunity for discussing the various lines of work on the floor of the convention is very important, adding greatly to a better understanding of the questions at issue, and increasing materially the attractiveness of our meetings. I think too much of the discussion is reserved for the various committees who have the several subjects in charge. I have no doubt they would be very glad to be relieved of some of the discussion, and have the greater part presented before the whole convention. The committee could then take the question under advisement, and if any additional facts are needed before submitting the report to the whole body, they could be called for. The convention, having heard the discussion of a question, would be better able to vote intelligently on the committee's report. It would not then be necessary, as is often the case now, for members continually to interrupt the chairman making the report by asking questions.

I would, therefore, plead for more discussion on the floor of the convention; and in order to give time for this would suggest that each referee in making his report be as clear and concise as possible, only reading such figures as are necessary to a clear understanding of the questions under consideration. It is not necessary to read the results obtained by the different analysts, especially when in many cases the members have the table in question before them.

I would also recommend to the consideration of the association the question of the adoption of a uniform method of statement of results on all the subjects which we have under consideration. It is hardly necessary for me to say how this would benefit us in our work. We all know how much difficulty we have when we are looking up results for comparison or getting up a compilation. We find results given in so many different ways and forms that it takes considerable work to figure them out, and, indeed, many results can never be used because sufficient data is not at hand to make calculations. I would also recommend that a change be made in the temperature at which our measuring apparatus is graduated. The temperature at present used, in nearly all cases, is 15.5° C. This is very much below room temperature, and often requires the cooling of solutions—a troublesome process, consuming considerable time. It seems to me that it would be of decided value to us if a temperature were adopted which would more nearly correspond to the temperature of our laboratories, which is usually about 20° C.

In conclusion I wish to renew a recommendation that was made last year by the last president, but which was not acted upon; that is to have the association consider the advisability of publishing Battle and Dancy's chemical conversion tables for the use and convenience of our members. These tables are now out of print, and I understand that the authors, if given proper credit for the same, are perfectly willing for the association to make such use of them as it may desire.

APPOINTMENT OF COMMITTEES.

The **President**. The appointment of committees is in order, and I will make the following appointments:

Committee to wait on the Secretary and Assistant Secretary of Agriculture: Messrs. Winton, Street, and Penny, Doctor Munroe, dean of Columbian University, and the president of the association.

Committee on resolutions: Messrs. Kilgore, H. B. McDonnell, and Jones.

Committee on nominations: Messrs. Wheeler, Frear, and Ross.

Committee on amendments to the constitution: Messrs. Patterson, Hills, and Magruder.

If there is no objection I will appoint the same number of committees on recommendations of referees as were named last year.

Committee A (phosphoric acid, potash, nitrogen, soils, ash, and insecticides): Messrs. Hartwell, Ross, Hopkins,[a] and Kilgore.

Committee B (dairy products, foods and feeding stuffs, sugar, and tannin): Messrs. Patrick, Withers, Van Slyke, and Kerr.

Committee C (food adulteration): Messrs. Leach, Munson, Penny, and Eaton.[b]

The PRESIDENT (continuing). The report of the referee on food adulteration is in order.

REPORT ON FOOD ADULTERATION.

By W. D. BIGELOW, *Referee.*

Heretofore the work on food adulteration has been largely done in committee, as you remember, and the action of the committee only has been reported to the association as a whole. It was necessary to do this because of the great amount of material that had to be gone over every year, much of which was attended to by correspondence, and even a small committee required two days at least, and sometimes more, to cover the ground. This year it was considered that we had reached the point where the work could be taken up in the association, and a number of reports have been prepared by subreferees. Unfortunately, in many cases the subreferees have not been able to present a report, owing to the fact that it was impossible to secure collaboration over the entire field. No report has been received for saccharine products—wine, beer, vinegar, baking powder, cereal products, infant foods, vegetables, tea, and coffee. Considerable work has been undertaken on cereal products, but, with the exception of one paper which will be presented here, nothing has been completed, ready to report. One paper by Mr. Chamberlain, relating to the separation of the nitrogenous constituents of flours, while prepared in connection with the work on cereal products, would be more properly presented in connection with the report on the separation of nitrogenous bodies, the study of the vegetable, meat, and milk and cheese proteids having been assigned to a special referee by the association two years ago. Also, work that was taken up by the referee on food adulteration a couple of years ago, and has been continued this year both by Mr. Grindley, on meat, and in the food laboratory of the Bureau of Chemistry on meat extracts, is to be reported at this meeting, but will probably come under the report on the separation of nitrogenous bodies also, although the work was begun in connection with the food methods, and has been largely conducted under that heading until the last year.

I would suggest that the various associate referees now take up the questions that have been assigned to them in their order. In regard to the subject of colors, which comes first on the list, I have a provisional report to submit from Mr. Berry, who has one of the most difficult subjects and has made a good beginning, but was unable to attend the meeting.

REPORT ON COLORS.

By W. G. BERRY, *Associate referee.*

Owing to the large amount of work necessary for the preparation of a complete and systematic résumé of the subject of colors in connection with food stuffs, and the failure of collaboration, I am unable to present a finished report on the subject, and desire therefore to defer the final report until next year. The following synopsis of the subject, with extracts from the incompleted work in hand, will give some idea of the manner in which the subject will be treated.

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[a] Mr. Grindley was later appointed in the place of Mr. Hopkins.

[b] Mr. Hird was later appointed in the place of Mr. Eaton.

It is suggested that the members of the association be invited to do what original work their time may permit of, on the points suggested below, and communicate their results to the associate referee during the ensuing year, for incorporation in the final report.

(1) Solubility of the coal tar and vegetable dyes in various solvents (ether, acetic ether, petroleum ether, methyl and ethyl alcohols, acetone, etc.) arranged according to their solubility—as easily soluble, difficultly soluble, and insoluble.

(2) Extractive values of the various solvents for dyes in neutral, acid, and alkaline solutions.

(3) Characteristics of the coloring matters as contained in fresh fruits, vegetables, wines, etc., with reagents and solvents, and dyeing properties.

(4) Testing such new schemes as may appear in the various chemical journals, or have appeared during the last few years.

SYNOPSIS OF REPORT ON COLORING MATTERS IN FOOD STUFFS.

For convenience in handling, the subject will be treated in a systematic arrangement under the following headings, viz:

I. Remarks in general on coloring matters liable to be present in food stuffs.
II. Colors to be considered.
  (*a*) Coal-tar colors, with their composition, commercial designations, and synonyms.
  (*b*) Vegetable colors; their botanical origin, common names, etc.
  (*c*) Mineral colors and pigments.
  (*d*) Organic lakes.
III. Harmless and harmful colors and dyes, so far as known.
IV. Commercial coloring compounds.
V. Grouping of food stuffs into general classes for examination, as follows:
  1. Butter, oils, milk, cheese, etc.
  2. Nitrogenous foods.
    (*a*) Flesh foods.
    (*b*) Canned vegetables.
    (*c*) Beer, malt liquors, etc.
  3. Starch foods (noodles, macaroni, etc.).
  4. Vinegar.
  5. Confectionery.
  6. Wines and spirituous liquors, fruit sirups, etc.
  7. Brandy, liqueurs, etc.
  8. Tea, coffee, spices, etc.
  9. Medicinal preparations.
  10. Cosmetics, soaps, etc.
  11. Miscellaneous material (tags, paper linings, etc.).
 Each class will be treated under the following heads:
  (*a*) Natural or artificial coloring.
  (*b*) Mineral or organic coloring.
  (*c*) General methods in detail for detecting the organic coloring matter, and, if possible, isolation of same for identification by tabular schemes.
  (*d*) Special tests for colors.
VI. Each of the eleven classes in detail under heads as given in V.
VII. Original work in behavior of coloring matters with solvents and reagents.
VIII. Tabular schemes for the identification of the isolated coloring matters.
IX. Addenda (what colors allowed, laws, etc.).

I.—REMARKS, ETC.

The field of color chemistry is very large, and the chemist in general analytical practice often neglects it owing to the vast amount of time and patience required for an intelligent understanding of the subject. Much good work has been done toward establishing methods for the recognition of coloring matters in food stuffs, but with the data at present available for such work the individual operator will find himself called upon to exercise his own judgment and apply his own experience to the subject-matter in hand.

The introduction of coal-tar dyes into the field of food stuffs complicates enormously the scope of the work to be covered in an examination of artificial coloring. Were it possible to limit the examination to a certain number of colors, the work would be materially simplified, but no sooner has the analyst perfected color schemes and tabulated reactions for the identification of such colors when new ones are found, and render previous schemes to a certain extent unreliable and necessarily subject to alteration.

Hence the most that can be hoped for is to establish general methods for determining whether artificial coloring has been resorted to, and if so, whether the coloring matter is of mineral, vegetable, or coal-tar origin. This fact having been established, special methods must be applied for the separation and identification of the individual coloring matters, often complicated by the presence of mixtures of several coal-tar colors, rendering color reactions useless until each of the separate colors has been isolated. The synopsis given is intended to be an outline of methods for the examination of food stuffs, to be elaborated and extended in the future.

The food chemist may be called upon for an examination of the coloring matters and commercial preparations used for coloring foods, and for the examination of foods for the presence of artificial colorings, identification of the added color, and the presence of poisonous metals due to the latter. He may also be called upon for an opinion as to the toxic or nontoxic character of the coloring matter, which not only requires an examination for poisonous metals present, as lakes and pigments, but in addition the determination of the presence of poisonous metals due to impurities, the identification of the color or colors, and a knowledge of the toxic effects of the pure color per se.

An examination, therefore, of the colors and preparations employed involves the identification of the heavy metals present, the nature of the color entering into the lake, whether vegetable or of coal-tar origin, its identification, and, in the case of mixed colors, their separation and identification, and, lastly, the presence of poisonous metals as impurities in the coal-tar dyes, or their presence as inherent constituents of the colors themselves.

Many of the colors contain metallic constituents, as in the triphenylmethane derivatives, especially the greens, such as malachite green, iodin green, ethyl green, victoria green, etc., which contain a double chlorid of zinc in combination with the organic matter. Some of the acridines and thiazines also contain zinc and naphthol green B contains iron.

In the preparation of the coal-tar dyes many become contaminated with arsenic, copper, zinc, tin, lead, etc., from the use of these metals and their salts during the process of manufacture. Sulphuric acid being used in some stage of the process of manufacture of nearly all the dyes, introduces the danger of arsenic being present. Hence all dyes used for artificial coloring, which in themselves are harmless, may become injurious from the presence of these poisonous ingredients, unless highly purified.

While alum lakes of the vegetable colors are in general harmless, the presence of lakes of tin, antimony, etc., should be carefully guarded against.

The vegetable colors should be examined for the presence of coal-tar dyes of a dangerous nature, and for other vegetable colors and substances and inert mineral matter added for cheapening.

The examination of the food stuffs themselves necessitates determining whether the coloring is due to natural or artificial means, and, if the latter, the color or colors must be separated and an examination of the material made for heavy metals.

In regard to the toxic effects of the purified coal-tar dyes, the chemist must rely upon the experiments and conclusions of investigators in this line of work, and should hesitate to express an opinion on a new color without a physiological test upon living organisms. The experiments of König, Weyl, and others should be consulted on this subject.

The methods of separating the colors from the material to be examined by the use of solvents involves the separation of the natural colors at the same time, and these must first be isolated before identification tests can be applied. This fact naturally necessitates a very comprehensive knowledge of the color reactions and the behavior of the natural colors with reagents. Complete data on this subject are as yet wanting.

The coal-tar dyes may, in general, be most satisfactorily separated by the double-dyeing method, with or without previous extraction with solvents, as the case may be, and in some cases may be identified directly by tests on the dyed fabrics, or more accurately by being removed from the fabric and subjected to purification. Great care and judgment must be exercised in applying the color reactions and arriving at a conclusion therefrom, as the presence of more than one dye, or some organic impurity, will lead to erroneous results. Several means of identification should be tried and a conclusion drawn only when the analyst is perfectly satisfied of the identity of the dye.

Many of the results obtained in the determination of a dye have been rendered ambiguous, owing to confusion of names applied to the same dye, and the analyst should always make a point of identifying any particular dye by giving the name of the original manufacturers, so that its composition may be known. As, for instance, Orange G might be either of the following, in default of a distinguishing mark, viz:

Orange G (C. J.). Anilin azo β naphthol.
Orange G (B). Anilin azo 2 naphthol, 6.8 disulphoacid sodium salt.
Orange G (H). Sulphanilic acid azo β naphthol sodium salt.

As a guide to the food analyst, a compilation is given below of the vegetable and animal colors, lakes, and pigments, and the coal-tar dyes, with their synonyms, which he may be called upon to examine. Many of the old vegetable colors have, of course, been discarded for the coal-tar dyes now so universally employed.

V.—FOOD STUFFS, ETC.

1. *Butter, oils, milk, cheese, etc.*

The natural coloring matter of milk is lactochrome, which may be precipitated from milk whey by nitrate of mercury in bright, red-orange, resin-like masses, softening at 100° C., freely soluble in water and hot alcohol, separating from the latter on cooling (Blyth). Cholesterin occurring in milk is soluble in hot alcohol, ether, carbon disulphid, and chloroform, and when treated with five parts sulphuric acid to 1 part water it is colored red, then violet (Moleschott's test); and with 1 part sulphuric acid to 1 part chloroform, is colored blood red, violet, or purple (Salkowski's test).

The coloring matter of some plants when eaten imparts color to milk of cows, as follows: Marsh marigold, yellow; dyer's madder, red; and true forgetmenot and knotweed, blue.

Certain bacteria develop a blue color in milk, which turns cherry red on addition of caustic alkalis, and returns blue on adding acids.

Added vegetable colors present may be derived from safflowers, carrot juice, marigold flowers, celandine, buttercups, turmeric, annatto, etc.

Coal-tar colors present may be spirit yellow R, nitrosamin red, aurantia, methyl orange, orange IV, phosphine, picric acid, Martins yellow, Mikado yellow, Mikado gold yellow, naphthol yellow S, amidoazobenzol, butter yellow (benzine-azo-dimethyl-anilin), naphthol yellow RS, Victoria yellow, coralline yellow, anilin yellow (amido-azo-benzol-hydrochlorid), acid yellow, diazo-benzine, etc.

Color preparations are carottine, a solution apparently of 1 part annatto in 4 parts of oil, turmeric taking the place sometimes of the annatto; orantia, a mixture of annatto, sodium carbonate, and water, etc.[a]

(a) Butter, fats, oils, etc. (fresh).

Added coal-tar colors will be present, with few exceptions, in the form of nonsulphonated bases, and will be indicated by the test of Bujard and Baier.[b] From 2 to 3 grams of fat are dissolved in 5 cc of ether and shaken with 5 cc of concentrated hydrochloric acid (1.125). If coal-tar colors are present the solution is colored decidedly red. "Butter yellow," however, colors hydrochloric acid (1.19) only slightly or not at all.

J. Vandriken[c] states that pure butter is completely decolorized by amyl nitrite. Proceed as follows:

1. *Amyl nitrite* (*acid*).—To 2 cc of filtered butter is added an equal volume of ether in a test tube. To this 6 to 10 drops of amyl nitrite are added and then shaken. Pure butter is decolorized at once. If unfiltered butter is used more reagent must be employed, and usually slightly warmed.

2. *Nitrous ether.*—To 2 cc filtered butter add 2 cc of ether, then 25 to 30 drops of nitrous ether, and shake vigorously. The decolorization takes place more slowly than with amyl nitrite. Carotin is not decolorized, saffron is only slightly altered, turmeric is not decolorized, and annatto is not decolorized.

Leed's method is given in detail in the Analyst.[d] In using this method, if the butter has become rancid the acid water from the petroleum-ether extract must be tested for reduced coal-tar dyes as given under milk.

Special tests for saffron, turmeric, marigold, and annatto are given by Martin;[e] for carotin, by Moore;[f] and for caramel, by Leach.[g] These methods are given in detail, and in various authorities special tests may be found for saffron, turmeric, carotin, and rocou.

(b) Milk (fresh).

Leach's method is given in detail in the Journal of the American Chemical Society.[h]

(c) Milk (sour).

Blyth's method is given in detail in the Analyst.[i] This method is important for detecting the presence of coal-tar dyes which have been reduced to colorless compounds due to fermentation.

(d) Cheese.

Extraction with hot alcohol for coal-tar dyes. Shake with petroleum ether for nonsulphonated and vegetable colors and proceed as with butter and milk.

(e) Schemes.

Some special color reactions tabulated for vegetable and coal-tar dyes occurring in the above class of foods.

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[a] Allen, Com. Org. Anal. 1889, **3**: 356.

[b] Hilfsbuch für Nahrungsmittelchemiker, p. 144.

[c] Chem. Ztg. Rep. 1901, p. 106.

[d] Vol. 13, p. 150.

[e] Analyst, **12**: 70.

[f] Analyst, **11**: 163.

[g] J. Amer. Chem. Soc., **20**: 207.

[h] J. Amer. Chem. Soc., 1900, p. 207.

[i] Analyst, 1902, p. 146.

The PRESIDENT. If there is no discussion the report of the associate referee on distilled spirits will be received.

REPORT ON DISTILLED SPIRITS.

By C. A. CRAMPTON, *Associate referee.*

Under date of June 10, 1903, the associate referee sent the following circular letter to six chemists whose names had been suggested as likely to undertake work on liquors:

> DEAR SIR: Concerning the collaborative work for the Association of Official Agricultural Chemists, upon distilled spirits, in which you have expressed a willingness to take part, you are informed as follows:
>
> The most important inquiries to be answered in judging of the character of a sample of distilled spirits are:
>
> (1) Does it contain ingredients deleterious to health? This is determined by the tests for furfurol, and adventitious, harmful additions, and, to some extent, by the estimation of fusel oil.
>
> (2) Is it genuine or factitious?—i. e., does it owe its flavor and color to a long period of aging in wooden vessels or has it been made from neutral or cologne spirits by adding coloring matter, or artificial flavoring, or both. The determination of the amount and character of the solid matter present usually affords the answer to this question, supplemented by tests for artificial coloring matter. By far the greater part of the spirits consumed in this country is of the latter class, which is, doubtless, as healthful as the former class, but a much cheaper product. The difficulties of discriminating between the two are, of course, greatly increased in the case of a mixture.
>
> On account of the small number of chemists who have offered to collaborate, I have not thought it advisable to send out identical samples. Samples of genuine spirits can be readily obtained in the shape of goods "bottled in bond," sold under a strip stamp which covers the cork of the bottle, and which gives the age of the spirits. Samples of factitious spirits can he made up by the analyst himself, or obtained in the shape of cheap rectified spirits. Genuine spirits are not all necessarily covered by a warehouse stamp; some very expensive whiskies are made by blending different varieties, which can not be done in bond.
>
> The nature and number of the estimations to be made I will also leave to the judgment of the individual operator. I would, however, recommend the careful analysis of a few samples, including all the usual determinations, rather than the application of a few estimations to a large number of samples. The determination of extract or total solid matter in a large series of samples of known purity would, however, be of value in establishing a standard, and I should like to have such results.
>
> In addition to the determinations described on pages 96 to 98, Bulletin No. 65, Bureau of Chemistry, U. S. Department of Agriculture, Provisional Methods, etc., I would recommend the determination of the copper-oxid reducing power and its expression as a ratio of the per cent of extract, also the solubility in boiling water of the extract (a method recently published by Lasche and Kiesslich in the Brewer-Distiller, 1903, 1), expressed in the same way. As an alternative method under "11.—Determination of coloring matter" besides the paraldehyde and ether solubility tests, I would be glad to have the fuller's earth absorption test subjected to examination (Crampton and Simons, Journal of the American Chemical Society, 1899, 21: 355), and will furnish a sufficient quantity of the fuller's earth used in this laboratory for that purpose, if notified.
>
> Please do not hesitate to ask for any additional assistance or suggestions concerning the work, if you so desire; the small number of chemists participating renders the work of correspondence comparatively light.
>
> Your report, containing results and suggestions, should be sent in not later than September 1.

Only two reports were received, namely, those of Messrs. Pilhashy and Sawyer. Both contain valuable analytical data and suggestions, and are given in full.

REPORT OF H. E. SAWYER, BOSTON, MASS.

A tabular statement of the results obtained in the collaborative association work on distilled spirits is appended. Report is made at this time on 15 samples—2 of "French" spirit, at proof strength, such as is used here for splitting genuine goods;

4 of our new rum, taken at time of drawing; 4 of our old rum, taken from original packages; and 5 of goods which were supplied by retail dealers in Boston upon demand for old pure rum for medicinal use. As all of the official analytical methods could not be tested, those which seemed likely to be most significant were tried, leaving those which promised less for later examination. The extract and fusel-oil determinations, not reported here, are under way. The methods tested are those for acids (free and combined), aldehydes, furfurol, and coloring matter. Concerning them the following comments and suggestions are made:

Acids, free and combined.

Occasionally samples of new rum contain more or less carbonic acid, which will raise the apparent percentage of acetic acid when decinormal soda and phenolphthalein are used for titration. Also, it is very difficult, when working close to a fermenting room, to protect solutions against the carbonic acid of the atmosphere. On this account baric hydrate was tried in place of a caustic alkali. So far as could be determined with a few analyses, it works very well. One's attention is called immediately to the presence of CO_2; and, in addition, certain differences are perceptible between the deportment of genuine and colored goods. Cold saponification is also being tried in place of boiling under reverse cooler. With a large number of samples, and limited desk room, it is believed that it will prove to be advantageous.

Aldehydes.

So far the writer has been unable to prepare aldehyde-free alcohol, and on this account colorations have been compared with an arbitrary scale made up by progressive dilutions of a fuchsin-methyl violet mixture. Accordingly the aldehyde determinations given can not be compared with those of other workers, but are included in the report chiefly because they show a distinct increase in the strength of the color reaction in old rum. Four samples only were scaled—2 new and 2 old; but more than a dozen other samples were examined comparatively, and without exception the older rum gives the stronger reaction.

The Simon trimethylamine-sodium, nitroprussid test for acetaldehyde will not work in rum, although in a proof spirit made from specially pure alcohol it was obtained.

Furfurol.

Furfurol, like the aldehydes in general, seems to increase auring storage, but it is not certain whether this is real or only apparent. I am inclined to believe in its reality, on account of the abundant production of the aldehyde in question during the charring of oak wood. It is intended to compare some uncharred packages with goods of the same age in charred barrels.

But it may be that there is an apparent difference, due to the presence in new rum of some material which, in part, obscures the furfurol reaction. On adding colorless c. p. anilin to colorless new rum a pronounced greenish-yellow coloration is at once developed. The same thing happens, though to a less extent, with some samples of commercial "proof spirit," such as the rectifiers use for splitting. The subsequent addition of acetic acid gives rise to the development of a pinkish coloration which is hard to estimate quantitatively, since it is obscured by the yellow, and in addition is very fugitive. In no case was the pink component of a new rum so determined, over 5 mg of furfurol per proof liter. If, however, the rum be redistilled before testing, the yellow tint is cut down materially, the pink component is at least doubled, and the red coloration is rendered far more permanent. (See samples 5 and 6.)

Coloring matters.

Both of the association methods for coloring matter were tried, using fuller's earth. With an earth which has been tested against several samples of known composition and with a good tintometer it is believed that a very close determination of added caramel, using the two methods in conjunction, may be made.

In connection with the matter of color, it is suggested that the extract of genuine aged spirits contains a good deal of tannin, whereas that of factitious liquors is not likely to. A test with ferric salts may be of value, and some work is being done on this point.

Fusel oil.

The official method has been inapplicable in my laboratory [Boston] during the summer on account of the temperature requirements. Therefore, the Allen-

Marquardt and Beckmann methods are being used, and ultimately it is hoped to compare the better of them with the official method.

The work, so far, has led to the conclusion that the acid and ester determinations are very significant. Their indications regarding samples 11–15 are fully in accord with the smell and taste of the liquors.

Results obtained by H. E. Sawyer.

No.	Sample.	Proof.	Acid.	Esters.	Aldehyde.	Furfurol.	Color left after fuller's earth treatment.	Color removed by ether.
			Gram per 100 cc.	*Gram per 100 cc.*	*Degrees.*	*Gram per 100 cc.*	*Per cent.*	*Per cent.*
1	Spirit	100.0	0.004	0.004	10	[a]0.0002		
2	....do	100.0	0.001	0.0004	10	[a]Trace.		
3	New rum	100.0	0.025	0.101		[a]0.0005		
4	....do	100.0	0.037	0.127		[a]0.0005		
5	....do	112.4	0.037	0.113	55	[a]0.0005 [b]0.0010		
6	....do	99.8	[c]0.076	0.084	55	[a]0.0005 [b]0.0010		
7	Old rum, 1902	100.5	0.054	0.061			34	33
8	Old rum, 1899	114.8	0.078	0.146			34	55
9	Old rum, 1896	115.3	0.078	0.171	65	[b]0.0045	34	55
10	Old rum, 1893	114.7	0.082	0.162	65	[b]0.0045	42	58
11	Rums bought from retail dealers.	100.0	0.042	0.088	55	[b]0.0022	11	None.
12		68.3	0.018	0.022	15	[b]0.0003	11	None.
13		89.7	0.007	0.032	30	[b]0.0003	11	None.
14		93.9	0.023	0.156			11	None.
15		86.7	0.011	0.150			11	None.
	[Proof spirit + caramel to old rum tint.]						11	None.

[a] Furfurol determinations made on original liquor.
[b] Furfurol determinations made on distillate.
[c] Much CO_2 present.

REPORT OF B. M. PILHASHY, CINCINNATI, OHIO.

The figures given in the following table are for 100 cc, 100 proof. The extracts in both samples agree closely as do the acidity and ethers. The ethers were determined first in the original goods, the acidity being first neutralized. The titration was made with phenolphthalein, and close readings were difficult. Determinations were then made by distilling as for volatile acids and ethers in wine. The difference is quite apparent and, in the writer's opinion, can hardly be due to the indicator.

Results by B. M. Pilhashy.

[Figures for 100 cc, 100 proof.]

Date of inspection and proof.	Extract.	In N/10 KOH.		Copper reduced.		In N/10 KOH.	
		Acidity.	Ethers.	In sample.	In extract.	Acidity after distillation.	Ethers after distillation.
	Grams.	*cc.*	*cc.*	*Grams.*	*Grams.*	*cc.*	*cc.*
February, 1900 (proof 108.6)	0.1401	11.0	17.6	0.0491	0.0855	4.7	8.1
October, 1900 (proof 109.0)	.1400	10.7	19.7	.0618	.0806	3.6	8.0

The difference between the copper reduced in the original liquor and in the extract of the same is noticeable. The reverse was expected, the difference to be attributed to aldehyde reduction.

These results are hardly to be believed, and the work will be repeated as soon as possible. Twenty-five cubic centimeters, or the extract from an equivalent amount, were boiled for 35 minutes with Fehling's solution. This method was tried to see if a reduction figure could be applied to the solid and the liquid parts separately Fusel oil was not determined, owing to the breaking of the apparatus.

The following are figures for new goods, i. e., high wines as barreled at 101 proof:

Results obtained on new goods, 101 proof.

Date.	Acidity (cc N/10 KOH).	Ethers (cc N/10 KOH).	Fusel oil.
			cc per 100 cc.
May 25	1.8	13.6	1.266
May 26	2.1	15.4	1.30
May 27	2.0	13.0	....
May 28	2.2	13.6	....
May 29	2.6	11.2	1.366
May 30	1.8	10.8	1.63
June 1	2.2	12.4	[a]1.26
June 2	2.6	11.3	1.13
June 3	2.2	12.4	1.20
June 4	2.2	12.2	1.23
June 5	2.3	12.4	1.76
June 6	2.0	10.6	1.25
June 7	2.2	14.2	1.28
June 9	2.4	13.0	1.38
June 10	2.4	11.6	1.00
June 11	2.8	6.3	1.11
June 12	2.4	7.6	1.10
June 13	2.0	7.2	1.36

[a] No copper reduction; in 2 other samples no copper reduction was obtained.

In the fusel oil determinations 21.42 cc was deducted as the figures for the $CHC_3 + C_2H_6O$ increase. This is taken from a bulletin and not verified.

It was the intention of the referee to report upon a line of experimental work which has been carried on in his laboratory for the past five years and which formed the subject of a preliminary communication to the American Chemical Society at its last annual meeting. Pressure of work in other lines, however, has prevented the completion and tabulation of results, which must be postponed for another year. This is regretted, as there are many points which will probably have a bearing upon methods of analysis when worked out, although the line of experiment is primarily in the direction of the establishment of composition standards, and is not a comparison or critical study of methods.

A careful study of the provisional methods prescribed by the association for the analysis of distilled liquors has convinced the writer of the desirability of modifying them in two or three of the principal determinations, and if this can be satisfactorily accomplished it will probably be sufficient progress for the present meeting.

The determination of alcohol, for instance, seems to be radically defective in not providing for a higher dilution of the liquor before distillation. As it stands, the method would give a distillate containing about 25 per cent of alcohol, which is entirely too strong and must inevitably lead to loss of alcohol. The amount taken for the determination should be cut down at least one-half, to 25 cc.

The referee has been accustomed to weigh rather than to measure the portion taken, using glass-stoppered weighing flasks. This is done in all cases where careful work is required, and only in this way can close agreement in results be attained.

RECOMMENDATIONS.

It is therefore recommended that the method as given in the provisional methods for the analysis of foods[a] be modified to read as follows:

(2) Determination of alcohol: Weigh, or measure (at 15.6° C.) into a distilling flask 20 to 25 cc of the sample, dilute with 100 cc of water, and proceed as directed on page 82. The sample of distilled liquor taken for the determination of alcohol is diluted more than in the case of wine, because of the errors attending distillates high in alcohol, errors due to evaporation and to making up to volume at temperatures varying slightly from 15.6° C. All measurements must be made at about that temperature.

The next determination, that of extract, seems to be a little misleading, as the solid residue left on evaporation by most spirits is too small in quantity to become sirupy in consistence. The following is therefore recommended as a substitute under "3. Determination of extract," page 96.

Weigh, or measure (at 15.6° C.) 100 cc, evaporate nearly to dryness on the water bath, then transfer to a water oven and dry at the temperature of boiling water.

The determination of ash may be allowed to stand.

In the determination of acidity, the recommendation of Mr. Sawyer for a change from sodium hydroxid to a barium hydrate solution, for titration, is deserving of consideration. While it has not been tested by the referee, the change is recommended on account of the advantages Mr. Sawyer found it to possess. Therefore, substitute the following, under "3. Determination of acidity," on page 96:

Titrate 100 cc (or 50 cc diluted to 100 cc if the sample is dark in color) with decinormal barium hydrate, using phenolphthalein as indicator. The number of cubic centimeters employed is multiplied by 0.006 for the acidity expressed in grams of acetic acid per 100 cc.

In the determination of fusel oil, a change should be made which has been adopted in all official methods abroad, viz, the distillation of the sample with alkali to remove free acid and saponify esters.

I would recommend the substitution of the following for the third paragraph of the description, page 96:

Add a small quantity of alkali to 200 cc of the sample under examination, and distill slowly, till about 175 cc have passed over; allow the distilling flask to cool, add 25 cc of water, and distill again till the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume (sp. gr. 0.96541 at 15.6°).

The method for the determination of ethereal salts, should be changed. The paragraph is vague, and in parts incorrect. Titration of the excess of mineral acid is not mentioned and no allowance is made for the subtraction of the volatile from the total acids. As the method stands, the calculation would give not only the excess of mineral acid added, but also both the free and combined acids in the sample.

Further, it might be questioned whether all of the ethereal salts distill at a temperature sufficiently low as to be found in the alcoholic distillate. I would recommend the substitution of the following, on page 98:

(9) Determination of ethereal salts: Neutralize the residue left after distillation in the fusel oil determination with N/10 H_2SO_4 and add an excess of 10 cc of the acid. Let stand five minutes, and make up to 200 cc. Titrate 2 portions of 25 cc each, using as indicators methyl orange in the first and phenolphthalein in the second. The difference gives the amount of alkali necessary to neutralize the organic acids in 25 cc of the sample. By subtracting from this figure the number of cubic centimeters of alkali required for the free acids and multiplying the result by 0.0088, the number of grams of ethereal salts (calculated as ethyl acetate) in 25 cc of the sample is determined.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 65, p. 96.

For next year's work I would suggest that the reporter make a comparative study of other methods of fusel oil determination, such as the Allen-Marquardt and Beckmann. The official (Roese) method is a very tedious, delicate operation, and the results are far from satisfactory. It is very desirable that some better method should be obtained.

About all the valuable information concerning distilled liquors which has appeared in the journals during the past few years will be found in the following references:

Beckmann: Ueber die Bestimmung des fuselgehaltes alkoholischer Flussigkeiten. Zeit. Nahr. Genuss., 1899, **2**: 709.

Beckmann: Neuerungen zur Bestimmung des fuselgehaltes alkoholischer Flussigkeiten. Zeit. Nahr. Genuss., 1901, **4**: 1059.

Schidrowitz: Composition of whisky. J. Soc. Chem. Ind., 1902, **21**: 814.

Zega: Examination of brandies from damsons and grape marc. Chem. Zeit., 1901, **25**: (75) 793.

Hewitt: The retarding influence of aldehydes on the maturation of potable spirits. J. Soc. Chem. Ind., 1902, **21**: 1896.

Mr. SAWYER. Mr. President, I would like to have two or three minutes for discussing the report. In my letter to the referee I said that there were certain determinations under way in my investigation which presumably would be completed before this meeting. It is of them that I wish to speak.

In the determination of fusel oil it was found impossible to carry out the Roese method, and I believe that it will not be practicable to use it in the average laboratory on account of its temperature requirements. The Beckmann method, to which the referee has referred, I have subjected to a preliminary test, and so far as can now be seen it is going to prove very satisfactory. I have not tested it to see how the results check, but understand that it has been tried in that respect by a number of German investigators whose figures have corresponded admirably. My test of the method was to determine whether it can be used in the ordinary laboratory at such a rate of speed as to make it suitable for routine work, and a single worker was able to make four determinations in an afternoon, which is better speed than could be obtained with the Roese method.

I suggested to Mr. Crampton that there was likely to be a marked difference between the behaviors of genuine and factitious liquors in regard to their reaction for tannin. This body is a characteristic ingredient of the extract of genuine distilled liquors which have been aged in oak casks. When the venders of factitious goods learn this they may begin to add tannin together with the burnt sugar which they now use for coloring, but for the present a test with ferric salts should show a distinct difference between aged and imitation goods.

There has been a tendency of recent years to hold furfurol responsible for those ultra-injurious effects of new liquor for which fusel oil used to be blamed. I believe that we shall have to abandon this doctrine and look for a new scapegoat. In the first place, new liquor contains less furfurol than old liquor does. In testing many samples of rum to see whether it disappears during the aging process, it was found that new goods contained a little, coming no doubt from the breaking down of pentosans in the molasses, which is our raw material. In rum from uncharred packages—that is, in plain oak casks—the amount was about the same as in new goods. Now, rum of this sort is not esteemed for drinking. The best liquor—that which is properly matured for drinking—is put away for years in oak packages which have been charred inside. And it is in this best grade of liquor, whose noxious effects are much less than those of the newer goods, that the proportion of furfurol is highest. Its source is obvious. It comes from the dry distillation of the oak wood which accompanies the charring of the barrels.

My own experience with furfurol—and I worked for three years in an atmosphere laden with its vapors—leads me to believe that it is not nearly so toxic as is generally supposed. Furthermore, I believe that it is to the more volatile constituents that new and factitious liquors owe their specially objectionable qualities. The disagreeable experiences which I have had in working with them have convinced me that we should set about determining what they are and formulating methods for their estimation.

Mr. Bigelow. There is no report on vinegar, but I wish to suggest a modification of the method for the determination of caramel as given in Bulletin No. 65, page 65, under "Detection of coloring matters." The presence of the precipitate obtained is not sufficient proof of the presence of caramel; its character must also be considered. I will draw up an amendment to the present method embodying this idea and submit it to the committee on recommendations.

The President. The subject of spices will now be considered.

REPORT ON SPICES.

By R. E. Doolittle, *Associate Referee.*

No work of particular interest on the methods for the analysis of spices has been done during the past year, so far as my readings and observations have extended. The referee reported that no one volunteered for collaboratory work on the subject in response to the circular sent out by him. Before receiving notice of my appointment as associate referee, samples of the various varieties of whole pepper had been obtained from the leading importing houses for the purpose of investigating their composition, more particularly with reference to the enforcement of certain statutes embraced in the food laws of the State of Michigan. During the year these analyses have been completed, following in general the methods adopted provisionally by the association, and the few comments made in this report are based upon my own observations during this work. There is one matter of importance in discussing the methods for the analysis of spices, and that is, many States have standards fixed by statute for one or more of the spices, these standards being based on the methods now adopted provisionally by this association. Further, as these methods were very carefully investigated and compiled by Mr. Winton, whom we all know to be our best authority on the subject, we should go very slowly in making any radical change.

In the determination of starch by the diastase method, an animal diastase manufactured by Frederick Stearns & Co. has been adopted in our laboratory in place of the powdered malt. This is a pancreatic diastase which gives no reduction on the Fehling solution, thereby eliminating that source of error and increase of work, and it is used exclusively in starch determinations at the University of Michigan. It is suggested that its use be investigated by the associate referee on spices for the coming year.

For determining the amount of copper reduced, I prefer weighing as cuprous oxid, as described by Munson under Methods for Determining Reducing Sugars.[a] The asbestos is prepared by cutting the best quality of woolly asbestos into fine pieces, digesting first with 1:3 hydrochloric acid and then with Fehling solution. The asbestos is packed into the Gooches by aid of a blunt glass rod, making a mat about one-fourth of an inch thick and thoroughly washed with hot water, then with 10 cc of alcohol, and then with 10 cc of ether. The Gooches thus prepared are dried in the oven at 100° C. for thirty minutes, allowed to cool one-half hour, and weighed. The cuprous oxid is collected, washed in the same manner with hot water, alcohol, and ether, and dried for thirty minutes at 100° C. and weighed after cooling for thirty minutes. The cuprous oxid may be dissolved off with hot dilute nitric acid and the same Gooch used for several determinations.

It is also suggested that in the determination of crude fiber the various kinds of filters and containers suggested in the provisional method[b] be thoroughly studied by collaborative work and the most serviceable plan reported. In our laboratory we conduct the boiling in 600 cc beakers covered with a watch glass, filter through linen after the acid treatment, and on weighed S & S blue ribbon filters after the alkali, washing thoroughly with hot water, then with a little alcohol, and then with ether, introducing the necessary correction for loss on treatment of the filter with the alkali, alcohol, and ether.

Mr. DOOLITTLE. As Mr. Winton is not here, I suggest that the discussion be postponed until he is present.

Mr. BIGELOW. There is no referee's report on meat, and as the only work done outside of our own laboratory is that reported by Mr. Grindley and is devoted chiefly to proteids, I suggest that his paper be read when the subject of nitrogen is discussed.

The PRESIDENT. Acting on this suggestion, the next report to be received will be that of the associate referee on dairy products considered from the standpoint of adulteration.

REPORT ON DAIRY PRODUCTS.

By ALBERT E. LEACH, *Associate referee.*

In my report I have chiefly to recommend a few methods to supplement those already included in the provisional methods as published in Bulletin 65 of the Bureau of Chemistry, with a brief statement of reasons, in some cases, for their insertion.

MILK PRESERVATIVES.

The hydrochloric acid and ferric chlorid method for detecting formaldehyde, not only in milk, but also in other food products using pure milk as a reagent, has been so thoroughly tested by over six years of constant trial that it would seem to be worthy of a place among our provisional methods. In point of delicacy it is far superior to the sulphuric acid test. I believe nothing but formaldehyde has yet been found that will give this peculiar and unmistakable color reaction.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 73. Proceedings of the nineteenth annual convention of the Association of Official Agricultural Chemists, p. 59.

[b] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 65, p. 154.

While salicylic and benzoic acids are not ideal milk preservatives, their presence is always to be looked for, and the following methods[a] for their detection in milk are submitted.

Benzoic acid.—Add 5 cc of dilute hydrochloric acid to 50 cc of the milk in a flask and shake to curdle. Then add 150 cc of ether, cork the flask and shake well. Break up the emulsion which forms, by aid of a centrifuge, or, if the latter is not available, extract the curdled milk by gently shaking with successive portions of ether, avoiding the formation of an emulsion.[b] Transfer the ether extract (evaporated to small volume, if large in bulk) to a separatory funnel and separate the benzoic acid from the fat by shaking out with dilute ammonia, which takes out the former as ammonium benzoate. Evaporate the ammonia solution in a dish over the water bath till all free ammonia has disappeared, but before getting to dryness add a few drops of ferric chlorid reagent. The characteristic flesh-colored precipitate indicates benzoic acid. Care should be taken not to add the ferric chlorid till all the ammonia has been driven off, otherwise a precipitate of ferric hydrate is formed.

Salicylic acid.—Proceed exactly as directed for benzoic acid. On applying the ferric chlorid to the solution after evaporation of the ammonia, the well-known violet color indicates salicylic acid when present.

These methods for salicylic and benzoic acids while especially applicable to milk, from which the ether extracts both fat and preservative, are useful also with modifications for many other food products. The extraction of the ether solution with dilute ammonia, whereby the preservative is removed, permits the subsequent recovery of the ether by distillation.

Detection of Color in Butter.

Annatto is very readily detected when present in notable amounts in butter, and when the straw color due thereto is concentrated permanently on a piece of filter paper, as in the test given below, there is no reason for mistaking it. With the azo and other coal-tar dyes the case is not so simple, and it would seem as if a number of the best methods for testing these colors in butter should be placed among the provisional methods, so that the analyst may base his opinion on the result of a variety of tests whether positive or negative. It is hardly safe to condemn a sample on a single test or method.

Method of the Massachusetts board of health for annatto.—Treat 2 or 3 grams of the melted and filtered fat (freed from salt and water) with warm, dilute sodium hydroxid, and, after stirring, pour the mixture while warm upon a wet filter; a hot funnel may be used to advantage. If annatto is present, the filter will absorb the color, so that when the fat is washed off by a gentle stream of water the paper will be dyed straw color. It is well to pass the warm alkaline filtrate two or three times through the fat on the filter to insure removal of the color. If, after drying the filter, the color turns pink on application of a drop of stannous chlorid solution, the presence of annatto is assured.

Color in Oleomargarin.

The question of natural color in yellow oleomargarin does not vitally concern the analysts in Massachusetts by reason of certain recent court decisions, according to which oleomargarin, even though resembling yellow butter, can be sold with impunity if it does not contain a foreign dyestuff—that is to say, if the color is due entirely to the natural oils which it contains. For this reason very little attention has been given to the presence of palm oil, which in some sections of the country seems to have caused considerable trouble.

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[a] Leach. An. Rep. Mass. State Board of Health, 1902, p. 475. Food and Drug Reprint, p. 23.

[b] A volume of ether largely in excess over that of the curdled milk has been found to be less apt to form an obstinate emulsion.

Oleomargarin without any color other than that inherent in the fat ingredients is now commonly found on the market, and, though devoid of dyestuffs, it rivals in depth of color butter which has been dyed. Frequent samples of this kind have been examined in our laboratory. On account of the processes to which cotton-seed oil and oleo oil are now submitted, with a view to enhancing their deep color, it is quite possible to obtain a very yellow oleomargarin without even resorting to the use of palm oil.

Most of the methods suggested for the detection of small quantities of palm oil in oleomargarin have not proved to be reliable. A number of such methods have been submitted by Mr. Ansil Moffatt, of Indianapolis, and while the writer has been unable, through lack of time, to personally test these methods, Mr. A. H. Gill, of the Massachusetts Institute of Technology, has tried them, and one of them, namely, the aluminum chlorid test, proved quite satisfactory in his hands. This test was carried out according to Mr. Moffatt's directions, as follows:

A solution of 10 per cent anhydrous aluminum chlorid in methyl alcohol is taken as a reagent. The clear filtered fat is put into a test tube, a few drops of the above reagent are added, and the test tube is heated with shaking until the solvent is evaporated and the alumina precipitated. The presence of palm oil is indicated by the color of the precipitate, which varies with the character of the oil contained in the mixture. Comparative tests had best be made on samples of known purity, both of pure oil and of mixtures. The following letter was received from Mr. Gill in regard to these tests:

> Regarding my experience in the use of the so-called "cookery test" of Mr. Moffatt, I am willing to make the following statements: Cotton-seed oil and oleomargarin containing 1 per cent or less of palm oil show a decided coloration when tested with aluminum chlorid in alcohol. Had I another test as decided as this, or had I made a careful series of tests with other deeply colored oils, I should be willing to accept it as conclusive proof of the presence of palm oil. From my experience in oil testing, I am unwilling to condemn an oil upon the results of one test, particularly if it be a color test. I have tried both the tests employing Nessler's solution and an alcohol and sulphuric acid test upon similar mixtures of palm with other oils without obtaining results which were satisfactory. I can not, therefore, indorse these tests.

PRESERVATIVES IN BUTTER.

In view of the fact that boric acid is commonly found in butter, a method for its detection therein should be provided. The following is submitted:

Detection of boric acid in butter.—Melt about 25 grams of the sample on the water bath, pour off the fat from the aqueous solution that settles to the bottom of the container, acidify the aqueous solution slightly with hydrochloric acid, and test in the usual manner with turmeric paper for boric acid.

FAT IN CHEESE.

During the past year a large number of cheese samples have been analyzed in the department of food and drug inspection of the Massachusetts board of health, and the following modification of the Babcock process for determining fat has been used to advantage:

Determination of fat in cheese—Lythgoe's modification of the Babcock method.—Weigh accurately about 6 grams of the sample in a tared beaker. Add 10 cc of boiling water and stir with a rod till the cheese softens and an even emulsion is formed, preferably adding a few drops of strong ammonia to aid in the softening and emulsionizing, and keeping the beaker in hot water till the emulsion is tolerably complete and free from lumps.

If the sample is a full-cream cheese, a Babcock cream bottle is employed. The contents of the beaker, after cooling, are transferred to the test bottle as follows: Add to the contents of the beaker about half of the 17.6 cc of sulphuric acid regularly used for the test, stir with a rod, and pour carefully into the bottle, using the remainder of the acid in two portions for washing out the beaker. Finally, proceed as in the Babcock test for milk. Multiply the fat reading by 18 and divide by the weight of the sample taken, to obtain the per cent of fat.

CONDENSED MILK.

It would seem that methods for the analysis of sugar-preserved condensed milk should properly be included under dairy products, and the following methods, which have been in constant use in Massachusetts for over eight years, are submitted:

Preparation of the sample.—Mix thoroughly by transferring the contents of the can to a large evaporating dish and working it with a pestle until homogeneous. Weigh 40 grams of the mixed sample in a 100 cc sugar flask, or transfer thereto by washing, and make up to the mark with water.

Total solids—Dilute a measured portion of the above 40 per cent solution with an equal amount of water, and transfer by a pipette 5 cc of the diluted mixture, corresponding to 1 gram of the condensed milk, into a tared platinum dish, which is allowed to remain in contact with the live steam of a water bath for at least two hours after the last traces of water have been evaporated to leave an apparently dry residue. Transfer to a desiccator, cool and weigh.

Ash.—Carefully ignite the residue from the total solids, cool and weigh.

Fat.[a]—Measure 15 cc of the above 40 per cent solution, corresponding to 6 grams of the condensed milk, into a Babcock test bottle. Fill nearly to the neck with water, add 4 cc of Fehling's copper solution, and shake thoroughly and rapidly, separating the precipitated proteids and fat by means of a centrifuge,[b] or the precipitate may be allowed to settle of itself, which it does more quickly in the cold. Withdraw the supernatant, sugar-containing liquid by means of a small-stemed pipette with a wisp of wet absorbent cotton twisted over the bottom to serve as a filter. Wipe off the cotton into the bottle on withdrawing the pipette. Give the precipitated proteids and fat two additional washings, as above, by shaking with water, separating the precipitate, and removing the washings with the pipette. If the precipitate is caked hard after centrifuging, use a stiff platinum wire as a stirrer. Finally, add water to an approximate volume of 17.5 cc and 17.5 cc of sulphuric acid, and continue the test as in the Babcock process of milk testing, multiplying the reading by 3 for the percentage of fat in the sample.

Proteids.—Dilute 5 cc of the 40 per cent solution, corresponding to 2 grams of the sample, to about 40 cc and add 0.6 cc of Fehling's copper solution. Nearly neutralize with sodium hydroxid stopping just short of alkalinity. Pass through a weighed filter paper, wash, dry at 100° C., and weigh. Burn the precipitate in a porcelain crucible, the difference between the weight of the dry precipitate and the weight of the ash being the weight of the proteids and fat. Expressing this in percentage and deducting the per cent of fat previously obtained, the result is the per cent of proteids.

Milk sugar.—Make up the filtrate and washings from the previous operation to 100 cc and determine the lactose either gravimetrically or volumetrically in this solution. If the volumetric method is used and the solution is of the exact strength directed above, milk sugar may be calculated as follows:

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[a] Leach. J. Am. Chem. Soc. 1900, **22**: 589.

[b] While the steam-driven centrifuge may be used for this, it is better to centrifuge in the cold, since the heat of the steam-driven machine cakes the precipitate so that it is harder to wash.

$$\frac{100 \times 0.067}{S \times 0.02} = L,$$

where L is the per cent of lactose and S the number of cubic centimeters of milk solution prepared as above necessary to reduce 10 cc of Fehling's solution.

Cane sugar.—Determine by difference, deducting the milk solids (milk sugar + proteids + fat + ash) from the total solids.

Adulteration.—The chief form of adulteration in condensed milk consists in the employment of skim milk in its manufacture. The nature and extent of the adulteration is not always apparent from the mere content of fat actually found in the sample, since this is to some extent dependent on the amount of condensation. Thus the most useful factor in judging of the purity of a sample is really the fat in the original milk. To calculate this it has hitherto been necessary to make a complete analysis of the sample to obtain the percentage of milk solids not fat. Divide this figure by an assumed standard for solids not fat (preferably basing this figure on the standard fixed by law), and the result expresses the "number of times condensed." Finally dividing the percentage of fat in the condensed milk by the "number of times condensed" the "fat in the original milk" is obtained.

We have recently adopted a simpler method for the calculation of fat in the original milk which involves merely the determination of fat and ash in the sample.[a] Assuming 0.70 per cent as the ash of pure milk, the factor representing the "number of times condensed" is found by dividing the ash of the condensed milk by 0.7. The "fat in the original milk," as thus calculated, is of course an arbitrary factor, and is useful only in deciding whether or not skimmed milk has been used in preparing the sample. By assuming the highest reasonable figure for the ash of natural milk, it is readily seen that the highest result is obtained for the fat in the original milk, and hence the benefit of any doubt is given to the manufacturer.

The ash of the condensed milk sample is conveniently obtained by evaporating to dryness 12.5 cc of the 40 per cent aqueous solution of the sample which forms the basis of the various determinations, this volume corresponding to 5 grams of the sample. The residue is then incinerated in the muffle, cooled and weighed. If the fat in the original milk as above calculated is found to be much below 3 per cent, there is evidence to show that skimmed milk has been used. This method of procedure is considered more accurate than that hitherto adopted for calculating the fat in the original milk, since the milk ash is a more constant factor than the percentage of solids not fat, on which the old method was based.

RECOMMENDATIONS.

I would recommend for provisional adoption the following methods: Under "Milk:"

The hydrochloric acid and ferric chlorid method for formaldehyde in milk.[b]—To 10 cc of milk in a porcelain casserole add an equal volume of concentrated hydrochloric acid containing 1 cc of 10 per cent ferric chlorid solution to each 500 cc of acid. Heat nearly to the boiling point over the free flame, holding the casserole by the handle and giving it a rotary motion to break up the curd. A violet coloration indicates formaldehyde.

Also the methods for salicylic and benzoic acids given in the opening of this report.

Under "Butter" the method of the Massachusetts board of health for annatto given in this report. And also the methods of Geisler, Low, and Doolittle for detection of colors, as follows:

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[a] An. Rep. Mass. State Board of Health, 1902, p. 475. Food and Drug Reprint, p. 23.

[b] An. Rep. Mass. State Board of Health, 1897, p. 558. Food and Drug Reprint, p. 20.

Geisler's method for azo colors.[a]—A few drops of the clarified fat are spread out on a porcelain surface and a pinch of fuller's earth added. In the presence of various azo dyes a pink to violet-red coloration will be produced in a few minutes. Some varieties of fuller's earth react much more readily than others with azo colors.

Low's method for azo colors.[b]—A small amount of the material to be tested is melted in a test tube, an equal volume of a mixture of one part of concentrated sulphuric acid and four parts of glacial acetic acid is added, and the tube is heated nearly to the boiling point, the contents being thoroughly mixed by shaking; the tube is then set aside, and after the acid solution has settled out it will be found to be colored wine-red in the presence of azo color, while with pure butter-fat comparatively no color will be produced.

Doolittle's method for azo dyes and annatto.[c]—The melted sample is first filtered. Two test tubes are taken and into each are poured about 2 grams of the filtered fat which is dissolved in ether. Into one test tube 1 or 2 cc of dilute hydrochloric acid are poured, and into the other about the same volume of dilute potassium hydroxid solution. Both tubes are well shaken and allowed to stand. In the presence of azo dye, the test tube to which the acid has been added will show a pink to wine-red coloration, while the potash solution in the other tube will show no color. If annatto has been used, on the other hand, the potash solution will be colored yellow, while no color will be apparent in the acid solution.

I would further recommend for provisional adoption under "Butter" the method for boric acid in the early part of this report, and under "Cheese," the modified Babcock method given for detecting fat.

And, finally, I recommend that the scheme for analysis of condensed milk as given in this report be provisionally adopted.

The PRESIDENT. Is there any discussion on the paper?

Mr. PATRICK. I am not sure that I understood the method proposed by Mr. Leach for determining proteids in condensed milk. Was it to precipitate with copper hydrate, dry, ignite, and take the loss as proteids——

Mr. LEACH. Weighing before ignition.

Mr. PATRICK. Is Mr. Leach sure that this method gives correct results? I am under the impression that it has been found lacking in accuracy, for the reason that the drying of the copper precipitate does not expel all the water.

Mr. LEACH. I think we have pretty thoroughly tested its accuracy, having used it in the analysis of many hundreds of samples during the past eight years. If you are careful in drying and keep the temperature limit between 100° and 105° good results are obtained, at least the error will be a very small one. The quickness of making the test also had something to do with our adoption of that method.

The PRESIDENT. The next subject for consideration is cocoa products.

Mr. BIGELOW. Mr. Eaton has submitted the first report on this subject, but, as has been the custom in regard to the other methods, it will be printed and distributed to the associates on food adulteration for informal criticism before it is submitted to the association for adoption as a provisional method.

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[a] J. Am. Chem. Soc. 1898, **20:** 110.

[b] J. Am. Chem. Soc., **20:** 889.

[c] U. S. Dept. of Agr., Bureau of Chemistry Bul. 65, p. 152.

The **President**. The report on preservatives is in order.

REPORT ON PRESERVATIVES.

By W. D. Bigelow, *Referee.*

Although the referee had the promise of considerable collaboration in the study of preservatives, and some work was begun, no definite results have been obtained. There is probably no field in connection with food chemistry which is in greater need of further study than that of the determination of preservatives.

The detection of some chemical preservatives is a very difficult matter because of the lack of reactions sufficiently delicate and characteristic to indicate their presence in the quantity in which they are used. It is certainly true that preservatives sometimes exist in foods which are reported by food laboratories as being free from them. On the other hand, there is a grave danger of error in the other direction, and it is believed by many that preservatives have been reported in samples in which they really did not occur, owing to the misinterpretation of certain class reactions and the giving of too much weight to reactions which were only sufficient to indicate "traces."

There is a tendency in some States to require by law that where preservatives are employed the kind and amount present should be stated on the label. In view of this fact it will probably be important in the near future to determine as nearly as possible the amount of preservatives present in foods. The methods that have been suggested heretofore for this purpose are inadequate.

During the last year considerable attention has been given to qualitative methods for the detection of formaldehyde and quantitative methods for the determination of salicylic and benzoic acid. It is intended to continue the work during the coming year and to extend it as far as the time and amount of collaboration available will permit.

The referee desires to emphasize the importance of this work and to request the collaboration of as many members of the association as possible.

The **President**. Mr. Winton is present and we shall be glad to hear the report on flavoring extracts.

Mr. **Winton**. I wish first to report on the committee's visit to the Secretary of Agriculture. I will take this opportunity to say that the Secretary received us very cordially, and agreed to address the meeting later in the day or perhaps early to-morrow. Knowing this, I hope we shall have a large attendance.

I have no written report to make on flavoring extracts, but will submit the following remarks:

REPORT ON FLAVORING EXTRACTS.

By A. L. Winton, *Associate Referee.*

Mr. Mitchell, the associate referee last year, outlined very good methods indeed, which have needed only slight changes. It is not necessary, I think, to go into those changes at the present time. There are, however, certain new lines of work which must be undertaken sometime in the near future. One subject in particular has been brought very forcibly to our attention during the past year by work done in the State of Michigan. It is not necessary for me to go into this subject in detail, as Mr. Doolittle is here and will give us full particulars of the case.

The matter referred to concerned lemon extract. It has been the custom of some manufacturers to prepare a cheap grade of lemon extract containing only a trace of lemon oil and but a fraction of the amount of alcohol prescribed by the United States Pharmacopœia. As the cost of alcohol in a good extract is several times as great as the cost of the other ingredients, the purpose of the manufacturer apparently is to reduce the amount of alcohol as much as possible. He does this by shaking the lemon oil with weak alcohol. The alcohol is too weak to dissolve any appreciable amount of the terpenes, but it does dissolve a certain amount of the citral and possibly other oxygenated constituents, and as a consequence the weak alcoholic liquid, after being separated from the undissolved oil, has a lemon odor. It has been the claim of these manufacturers and certain chemists that this "washed out" extract is equal or superior to that made by the old process, because the terpenes, which they regard as valueless, are eliminated.

The supreme court of Michigan has rather forced us to consider this matter, because while they have accepted the United States Pharmacopœia as the standard they have ruled that manufacturers have the privilege of improving on the processes of the Pharmacopœia. It is my opinion—and in this matter I have worked in thorough accord with Mr. Doolittle, of Michigan—that the citral is not by any means the only valuable ingredient of lemon extract, but that the terpenes also have decided value. I think those who have tried both the washed out and the genuine U. S. P. extracts in food products will agree that the flavor is somewhat different. While the terpenes of themselves may not be so aromatic as the citral, still in combination with the citral they give a blended flavor which is the true flavor of lemon. Either one by itself is not sufficient. It has been my custom to compare the part played in the extract by limonene, the chief terpene of lemon oil, to the low pipes of the organ. These low tones of themselves are not particularly agreeable, but combined with the high tones they produce the indescribable depth and grandeur of organ music. So in the case of lemon extract, the limonene alone is not highly aromatic, but blended with the citral it contributes strength or body to the flavor. While it is my firm belief, based upon considerable investigation, that the value of an extract is not determined by the citral alone, at the same time I think it is highly desirable that a good method for determining citral be decided upon, if for no other purpose than to assay extracts made with a washed out oil, which is even a greater fraud than making extracts by the washing out process. Mr. Doolittle investigated this matter thoroughly, and I hope he will talk to us on the subject.

There is another line of work which must be taken up eventually, and that is the examination of the mixtures of so-called fruit ethers made in imitation of various fruit flavors, such as strawberry, raspberry, and banana.

The cheap soda-water sirups are very largely made out of these mixtures of ethers with coal-tar dyes and other ingredients, many of them containing no genuine fruit extract or fruit juice whatever. Anyone who has had any experience with ice cream or soda water purchased at watering places knows that by the palate alone one can very quickly distinguish between the artificial and the genuine. What we need now are better chemical methods for their detection. Of course these artificial extracts of strawberry or raspberry must be colored in order to pass for the true fruit sirups. Almost always the artificial color can be detected and that at once brands the article as not being made entirely from the fruit; but the difficulty is that the genuine products are also colored so the presence of an artificial coloring matter does not tell us whether the product is purely an artificial mixture or whether it is a genuine fruit product which has been colored to improve its appearance.

Our methods for vanilla extracts, with slight changes, are quite satisfactory. We have certain difficulties to contend with, to be sure, but many of them are being overcome by analytical research. For example, there is no way of determining whether the vanillin in vanilla extract is from the genuine bean or from the synthetic product. The determination of vanillin is usually of almost no value in determining adulteration except in rare cases where none at all or an abnormally large amount is present. If prepared according to the United States Pharmacopœia vanilla extract contains about 0.2 per cent of vanillin while some of the cheapest extracts examined contained 0.6 per cent. In most cases the examination for coloring matter aids greatly in discriminating between genuine and artificial extracts. On the whole, our methods for examining vanilla extract are fairly satisfactory, as, indeed, are the methods for lemon extract with the exception of the one point—the determination of citral.

Mr. **Doolittle.** Mr. Winton has quite thoroughly covered the chemistry of the lemon extract controversy. The facts of the particular case referred to were as follows: When this subject came up in the courts of Michigan we were confronted by expert testimony from the State universities of Michigan and Wisconsin, and it developed in the course of the trial that in those States a product called terpeneless extract of lemon was being made. That is, the manufacturers claimed to extract from lemon oil only the flavoring properties, leaving the hydrocarbon portion behind. They claim that by agitating a 5 per cent solution of oil with a dilute alcoholic solution for a given length of time, and repeating the agitating after standing, all the citral and other flavoring constituents of the oil are extracted. This case was carried to the supreme court of the State of Michigan, and in the opinion handed down the court held as follows: That the legislature had in mind, when the food law was formulated, the formula of the United States Pharmacopœia for the manufacture of lemon extract, and that this formula is the standard for lemon extract, but that this does not prevent any manufacturer from producing an extract by any improved process, provided that the extract contains all the valuable constituents, i. e., all the flavoring constituents of a 5 per cent solution of lemon oil. In this particular case the manufacturers admitted that they used only a 5 per cent solution of lemon oil, and as experiments show that it is impossible by any chemical process to remove all the citral and other flavoring constituents, producing a perfectly tasteless terpene, they admitted that the extract in question was not up to the standard as defined by the court, and so pleaded guilty. But they now claim to use a 10 per cent solution of lemon oil and by treating this by the method described to remove from the increased amount of oil enough citral and other flavoring ingredients to equal those present in a 5 per cent solution of the oil.

Another curious fact brought out during the trial illustrates what Mr. Winton has said, i. e., the manufacturers are making a terpeneless extract for Michigan, dissolving the residual oil in alcohol and selling it in Wisconsin for a 5 per cent solution, thus complying with the U. S. P. formula as is required in Wisconsin, and I do not believe that the analyst can detect the exhausted product. Our methods for the analysis of lemon extract depend upon the amount of hydrocarbons in the solution. What confronts us in Michigan, therefore, is the need of a method for determining the amounts of citral in such alcohol solutions as lemon extract and also a method of qualitatively and quantitatively, if possible, determining the other oxygenated constituents of the oil that go to make up the flavor of the extract and are characteristic of lemon oil.

The PRESIDENT. Would it not bring relief if the standards were changed so as to conform to those of the adjoining States?

Mr. DOOLITTLE. That would not help us, as the manufacturers use this exhausted oil and still come up to the requirements of such standards.

Mr. LEACH. In Massachusetts we have been interested in investigating the people who have used the so-called terpeneless extracts without any oil whatever, and one manufacturer has taken the bull by the horns by trying to conform to our other statute which compels labeling all compound goods with the percentage of ingredients, and he words his label thus: "Avoid lemon extracts containing hydrocarbon compounds because they are apt to deteriorate. Take only extracts that have none of those objectionable ingredients. This is warranted to contain none."

Mr. DOOLITTLE. That is one of the strongest claims made and it is sustained by expert authority in Wisconsin and also by Doctor Vaughan, of Michigan. Not only that, but it is claimed that the use of strong alcohol is a very great detriment in the lemon extract because alcohol tends to curdle products containing milk or eggs.

Mr. FISCHER. I examined a lemon extract recently in Wisconsin which, when assayed by the precipitation method, gave 100 per cent lemon oil, but when assayed by the polarization method it showed only 15 per cent. It seems to have consisted of about 15 parts of so-called "robbed" oil, dissolved, and 85 parts of cotton-seed oil. Evidently the provisional methods are very much at fault in such a case. In assaying the oil of lemon it appears to me, without claiming to be an expert, to be extremely difficult to determine the amount of citral in the oil itself, and next to impossible to determine the amount of other oxygenated products; and when it comes to a 5 per cent solution the methods fail completely.

Mr. BROOKS. Does not lemon oil contain naturally these oxygenated products, and if the Pharmacopœia directs that the extract of lemon shall be a solution of oil of lemon, is there any way of evading that requirement? If the law is so worded as to require a solution of the whole oil, what claim can be made for a lemon extract made by using a robbed oil, or even the citral oil?

Mr. LEACH. I think there is a means of testing the separated oil, and am inclined to think there would be a difference in refraction between the so-called "robbed" oil, or, as we call it, the "float" oil, and the pure oil.

At 12.40 the convention adjourned until 1.30 o'clock p. m.

THURSDAY—AFTERNOON SESSION.

After some instructions to committees B and C on recommendations of referees, the president called for the report of the referee on foods and feeding stuffs, which was submitted by Mr. Van Slyke, acting for Mr. Fuller.

REPORT ON FOODS AND FEEDING STUFFS.

By F. D. Fuller, *Referee.*

In reply to a circular letter sent out inviting cooperation in the work for 1903, eleven chemists promised to assist. To these men the following instructions were sent:

Instructions for Work on Feeding Stuffs for the A. O. A. C., 1903.

Two samples are sent out this year for work on feeding stuffs.

No. I. Distiller's grains.

No. II. Wheat bran.

The following determinations are to be made upon both samples: Moisture, fat, and crude fiber.

Moisture is to be determined by the official method, Bulletin No. 46, Revised Edition, Department of Agriculture, page 23, or according to the method used in your laboratory. In reporting results state method employed.

Fat (ether extract) is to be determined by the official method, Bulletin No. 46, page 23, and also by the Dormeyer method. A description of the latter method is sent herewith.

Crude fiber is to be determined by the official method, Bulletin No. 46, page 26, by König's method, and also by using a modification of König's method. Inclosed please find a description of König's method, and also one of the proposed modification.

Please report results at your earliest convenience.

Yours truly,

F. D. Fuller, *Geneva, N. Y., Referee.*
L. H. Smith, *Urbana, Ill., Associate referee.*

It gives me pleasure to acknowledge the kindness of the J. W. Biles' Company, of Cincinnati, Ohio, who furnished a supply of Biles's Fourex distiller's grains for the use of the referee. The bran was taken from a lot in use at the New York experiment station, and previous to grinding 8.81 per cent of water was removed at 60° C.

Estimation of Moisture.

The moisture was not determined as a matter of investigation of methods, but simply to make it possible to calculate the other determinations to a water-free basis.

Table I.—*Moisture determination.*

Analyst.	Method of drying.	Distiller's grains.	Bran.
		Per cent.	*Per cent.*
E. B. Holland, Massachusetts	Glycerin and water oven, 100° C	7.38	7.09
H. J. Warner, U. S. Department of Agriculture.	Water oven, 5 hours	6.11	4.18
Do	Water oven, 22 hours	6.72	4.21
Do	In vacuo, 5 hours, 80° C	6.24	4.90
Do	In vacuo, 5 hours, 100° C	6.65	5.70
H. H. Hanson, Maine	Steam oven, 5 hours, 98° C	6.40	4.26
C. D. Howard, West Virginia	In vacuo, 2 hours	6.97	5.43
J. A. Bizzell, New York (Ithaca)	Official method	6.99	5.73
L. H. Smith and E. M. East, Illinois	Glycerin and water oven, in hydrogen, 5 hours, 104-105° C.	4.39	5.06
A. W. Bosworth, Rhode Island		7.04	4.94
C. W. Mudge, New York (Geneva)	Steam oven, 10 hours, 98° C	6.91	4.98

ESTIMATION OF ETHER EXTRACT.

According to the recommendation of the referee on foods and feeding stuffs for 1902, we have compared the Dormeyer method with the present official method of fat extraction.

In the Chemiker Zeitung[a] Beger gives some results obtained by digesting the materials in pepsin solution previous to extracting the fat. In all cases an additional quantity of fat was obtained after the pepsin treatment, the increase being greater when using materials rich in protein.

This process was first used by Dormeyer[b] and later studied by Nerking,[c] who argued that the ordinary extraction with ether could not remove all the fat, because it was chemically combined with the albuminoids "Fetteiweissverbindungen," and the compounds must be broken up and the fat liberated before it could be completely removed. Following is a description of the Dormeyer method:

Three to 5 grams of substance are digested with 1 gram of pepsin, Merck (this is found to be free from ether extract), dissolved in 480 cc water and 20 cc of 25 per cent hydrochloric acid at 37°–40° C. for twenty-four hours. The residue is then filtered in a Gooch crucible, washed several times with cold distilled water, and after drying is extracted with ether in the usual way. The filtrate must be shaken out with ether several times and the extract from this united with that from the dried residue.

TABLE II.—*Ether-extract determination.*

Method and analyst.	Distiller's grains.		Bran.	
	Original sample.	Water-free.	Original sample.	Water-free.
OFFICIAL METHOD.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Massachusetts	10.67	11.52	5.05	5.53
	10.67		5.18	
			5.19	
H. J. Warner, U. S. Department of Agriculture	10.13	10.84	5.12	5.43
	10.13		5.20	
	10.14		5.20	
H. H. Hanson, Maine	10.65	11.29	4.90	5.16
	10.49		4.97	
J. A. Bizzell, New York (Ithaca)	10.84	11.61	5.25	5.53
	10.75		5.17	
L. H. Smith and E. M. East, Illinois	10.44	10.85	5.22	5.47
	10.18		5.19	
	10.30		5.18	
	10.44		5.17	
	10.51			
A. W. Bosworth, Rhode Island	15.92	[d]17.22	9.51	[a]10.05
	16.10		9.59	
C. W. Mudge, New York (Geneva)	10.97	11.90	5.23	5.49
	11.13		5.19	
	11.15		5.25	
Average		11.33		5.43

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[a] 1902, vol. 26, No. 11.

[b] Pflügers, Archiv. f. Physiologie, 1895, **61**: 341; ibid., 1896, **65**: 90.

[c] Pflügers, Archiv. f. Physiologie, 1901, **85**: 330.

[d] Result omitted from average.

TABLE II.—*Ether-extract determination*—Continued.

Method and analyst.	Distiller's grains.		Bran.	
	Original sample.	Water-free.	Original sample.	Water-free.
DORMEYER METHOD.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Massachusetts	12.36	13.26	5.70	6.27
	12.19	....	5.96	....
H. J. Warner, U. S. Department of Agriculture	10.91	11.76	5.05	5.33
	11.03	....	5.09	....
	11.07	....	5.11	....
H. H. Hansen, Maine	11.77	11.87	4.85	5.07
	10.46	....	....	....
J. A. Bizzell, New York (Ithaca)	11.49	12.32	5.14	5.55
	11.43	....	5.31	....
L. H. Smith and E. M. East, Illinois	8.42	10.01	4.50	5.37
	8.87	....	4.99	....
	10.20	....	5.44	....
	10.77	....	5.48	....
Average	....	11.84	....	5.52

NOTES AND COMMENTS OF ANALYSTS.

E. B. Holland.—After the digestion with pepsin the solution was filtered through linen and washed to a total volume of about 800 cc. This combined filtrate and washings were shaken out with ether three times in a separatory funnel, which cleared the solution considerably. The extract previous to the evaporation of the ether was of a white gelatinous appearance. The residue in the filter, after drying, was inclosed in a capsule and extracted as in the official method.

H. J. Warner.—Found the Dormeyer method long and tedious.

H. H. Hansen.—Much difficulty found in filtering after the digestion with pepsin. Much more time was consumed and greater care required with this method than with the official method. There is, moreover, much greater chance for error.

L. H. Smith and E. M. East.—A second extraction of 16 hours by the official method was made in every case. As only an insignificant amount of extract was obtained, we may conclude that 16 hours is ample time for the extraction. The results by the Dormeyer method are not satisfactory, and most of the results appear to be too low, as compared with those by the official method. The method, on the whole, does not appear to be practical.

COMMENTS OF REFEREE.

A glance at the foregoing table will show that the results obtained by the Dormeyer method are a little higher than those secured by the official method, greater differences being observed in the results from distillers' grains, a food which contains over twice as much crude protein as wheat bran, a fact which might readily lead one to suppose that some of the fat was combined with the albuminoids, as maintained by Nerking, and only needed a digestion with pepsin to bring it into a readily ether-soluble condition. However, the question which arises at this point is this: Is the additional extract obtained by the Dormeyer method a positive increase in the percentage of crude fat contained in the material under examination? We all know that the ether extract of the official method is already much too high, as it contains bodies which can not come under the head of crude fat, and that by extracting with petroleum ether we secure an extract which more nearly represents the fat content of foods. Therefore we do not care to increase the amount of ether extract unless we are absolutely sure of making an addition of fatty bodies alone. Beger[a] made a few qualitative tests upon the extract obtained by the Dormeyer method and found present a fat which responded to the acrolein and refractometer tests, "fat spot," saponification, etc., but he made no examinations to prove the purity of the extract or to settle beyond doubt the exact nature of the fat present.

Had time permitted, it was the intention of the referee to make these particular points a subject of research. Your referee believes that valuable time is lost in preparing samples, sending them to various men for analysis, and attending to the necessary correspondence pertaining to the same, which otherwise might be used by the referee and his associate in conducting systematic research in the branch of work to which they may be assigned by the association. Therefore, I would recommend that next year the referee and associate devote a part of their time to research work in connection with the points mentioned above.

ESTIMATION OF CRUDE FIBER.

The official method for the estimation of crude fiber originated from the demand for a fiber which contains only a very small quantity of nitrogen. But since we have considered the pentosan content of foods it has been proven by König and many other analysts that the crude fiber obtained by the official method contains pentosans and in some materials they are present in considerable quantity. Since the official method does not give us a pentosan-free fiber it was the object of the referee to seek for a new method by which such a fiber may be prepared.

In 1900 the attention of the association was directed to a method by J. König[b] for the estimation of pentosan-free crude fiber. In 1901 Mr. Fraps presented a paper before the association in which he showed that the König method is superior to the official method, in that it yields a fiber which is practically free from pentosans. But he also showed that in the case of cotton-seed meal and other materials rich in nitrogenous bodies, the fiber obtained is much too high. Acting upon his suggestion, a study of the König method was taken up by the referee in 1902, who corroborated the results obtained by Mr. Fraps and showed, furthermore, that the König method, while removing the pentosans, does not yield a fiber low in nitrogen. Therefore he recommended that the present referee make a further study of this method with the additional treatment of the fiber with alkali, as in the official method. The König method was thus modified, and the results in Table IV are obtained by using the official, König, and König modified methods. Following is a description of the König method and also the modification, with such precautions and suggestions as seem necessary.

THE KÖNIG METHOD FOR CRUDE FIBER.

(*a*) *Glycerol-sulphuric acid.*—Determine the specific gravity of the glycerol by means of a picnometer or specific-gravity balance. A hydrometer can not be used, as on glycerol its readings would not be correct. The per cent of glycerol may be calculated by the following table:

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[a] Chem. Ztg. 1902, No. 11.

[b] Analyst, 23: 47.

TABLE III.—*Calculation of glycerol from specific gravity.*

Specific gravity at 15.5° C.	Glycerol.	Specific gravity at 15.5° C.	Glycerol.
	Per cent.		*Per cent.*
1.2674	100	1.2460	92
1.2647	99	1.2433	91
1.2620	98	1.2406	90
1.2594	97	1.2380	89
1.2567	96	1.2353	88
1.2540	95	1.2327	87
1.2513	94	1.2300	86
1.2486	93		

Should the temperature of the specific gravity determination not be 15.5° C, a correction may be made by adding 0.00058 for each degree above the temperature.

Dilute the glycerol to 86 per cent, or 1.230 specific gravity, and make up a solution containing 20 cc sulphuric acid (1.84 specific gravity) per liter of the glycerol-acid mixture.

(*b*) *The determination.*—Place 3 grams of substance in a 500 cc Erlenmeyer flask, add 200 cc glycerol-sulphuric acid, and connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Heat to boiling, and boil very gently for an hour, shaking the flask from time to time so as to wash down any particles. The boiling should take place in such a manner that only a few drops of water are condensed. Prepare a thin layer of asbestos in a 2-inch Hirsch funnel, and on this place a perforated platinum disk. Filter the glycerol on this, using a suction pump. The mixture should be shaken well before it is poured on the filter, and each flask should have the full benefit of the suction pump when the filtration starts until the glycerol begins to come through. Wash with hot water, with alcohol, and with a mixture of equal volumes of alcohol and ether. The alcohol and ether remove, besides fats, certain products not soluble in water formed by the action of the glycerol acid. Transfer to a platinum dish, dry, weigh, and incinerate completely. The loss in weight is crude fiber.

(*c*) *Precautions.*—A primary necessity is to have the glycerol of the required density. A comparatively slight variation in density will cause an increase or decrease in boiling point, with a corresponding decrease or increase in the crude fiber.

In filtering, considerable time is gained by using a flat-bottomed porcelain funnel, known in German laboratories as a "Nutsche." Dilute the hot glycerol-acid mixture with 300 cc of boiling water and filter through linen.

(*d*) *Modification of König's method.*—Treat as in the König method and after washing transfer the residue to an Erlenmeyer flask, using 200 cc of hot 1.25 per cent sodium hydrate solution. Attach a condenser and boil gently for 30 minutes. Filter and wash with hot water. Transfer the residue to a Gooch crucible, dry, weigh, and incinerate completely. The loss in weight is crude fiber.

TABLE IV.—*Crude fiber determination.*

Method and analyst.	Distiller's grains.		Bran.	
	Original sample.	Water-free.	Original sample.	Water-free.
OFFICIAL METHOD.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
E. B. Holland, Massachusetts	14.36	15.61	11.02	11.70
	14.79	....	11.13	....
	14.34	....	10.69	....
	14.34	....	10.75	....
	....	....	10.75	....
	....	....	10.90	....
H. J. Warner, U. S. Department of Agriculture	12.99	13.92	10.62	11.07
	12.96	....	10.58	....
	13.15	....	10.43	....
H. H. Hanson, Maine	14.09	15.05	10.95	11.43
C. D. Howard, West Virginia	14.25	15.34	11.08	11.68
	14.29	....	11.02	....
J. A. Bizzell, New York (Ithaca)	13.83	14.93	11.33	11.96
	13.95	....	11.20	....
L. H. Smith and E. M. East, Illinois	14.22	15.19	12.14	12.78
	14.33	....	12.22	....
	15.02	....	12.03	....
A. W. Bosworth, Rhode Island	9.57	[a]10.13	6.97	[a]7.42
	9.27	....	7.12	....
C. W. Mudge, New York (Geneva)	13.65	14.66	11.01	11.59
Average	....	14.96	....	11.74
KÖNIG METHOD.				
E. B. Holland, Massachusetts	19.97	22.35	11.51	12.49
	21.43	....	11.69	....
H. J. Warner, U. S. Department of Agriculture	17.20	18.44	11.75	12.20
	17.20	....	11.48	....
	17.34	....	11.62	....
H. H. Hanson, Maine	19.78	21.13	14.97	[a]15.64
C. D. Howard, West Virginia	18.06	19.08	11.98	12.27
	17.73	....	11.52	....
	17.45	....	11.31	....
J. A. Bizzell, New York (Ithaca)	15.58	16.54	11.76	12.50
	15.18	....	11.80	....
L. H. Smith and E. M. East, Illinois	17.49	18.87	13.70	13.18
	17.70	....	12.27	....
	17.51	....	11.56	....
A. W. Bosworth, Rhode Island	....	....	11.55	12.15
C. W. Mudge, New York (Geneva)	19.45	22.21	11.50	12.61
	21.90	....	12.46	....
Average	....	19.73	....	12.49
KÖNIG METHOD, MODIFIED.				
E. B. Holland, Massachusetts	11.70	12.73	9.97	10.82
	11.88	....	10.13	....
H. J. Warner, U. S. Department of Agriculture	11.03	11.88	9.55	9.96
	11.07	....	9.52	....
	11.26	....	9.40	....
J. A. Bizzell, New York (Ithaca)	11.44	12.36	10.39	10.94
	11.56	....	10.22	....
L. H. Smith and E. M. East, Illinois	11.66	12.17	10.24	11.24
	11.61	....	11.09	....
A. W. Bosworth, Rhode Island	14.48	[a]15.58	9.72	10.23
C. W. Mudge, New York (Geneva)	10.75	11.48	9.52	9.70
	10.62	....	8.92	....
Average	....	12.12	....	10.48

[a] Results omitted from average.

NOTES AND COMMENTS OF ANALYSTS.

F. B. Holland.—In the official method both filtrations were made through linen and the residue finally transferred to a platinum Gooch crucible and dried at 100°C. With the König, and modified König, methods the acid solution was diluted with an equal volume of hot water and filtered through linen.

H. J. Warner.—The König method while extracting pentosans evidently leaves a large amount of nitrogenous matter unextracted. The modified König method seems to give the best results.

H. H. Hansen.—König's method did not give satisfactory results in any case. Difficulty was encountered in filtering. Both Gooch and linen filters were tried using suction pump and diluting the acid mixture with hot water. In some cases the filtration stopped entirely.

C. D. Howard.—In filtering, a thin pad of asbestos upon a 2-inch Hirsch funnel seemed to give the best results. Filtration was found to be very materially hastened by washing fiber back into a beaker once or twice, replacing any asbestos that may have been removed. Dilution of the hot mixture before filtering was found to be helpful in the case of wheat bran, but gave no advantage with distiller's grains. The washing with alcohol is tedious, and the extent to which it is carried appears to have a material influence upon results. The present official method seems preferable to the König, apparently giving results nearer the truth, besides being quicker and easier of operation.

J. A. Bizzell.—In the fiber determinations the last filtration was made on linen, the residue transferred to a platinum dish and evaporated to dryness. This method was found preferable to filtration on a Gooch. In the König method the mixture was diluted with alcohol and filter through linen. The filtration was very slow.

L. H. Smith and E. M. East.—When asbestos was mixed in the sample before boiling, and the filtration made on hardened filter paper in a 4-inch Buchner funnel, the acid mixture being diluted with 300 cc of hot water, filtration was obtained in three minutes. Other methods took from ten to twelve hours. In comparing the two methods, König and official, we have to consider besides the point of accuracy those of convenience and expense. In regard to convenience the relative length of time required for the operation is about the same. As to expense, the cost of reagents is greatly increased by the use of glycerol and ether in the König method. The results by the modified König method are all lower than by the official method, showing a further solvent action by the alkali.

A. W. Bosworth.—In the König, and modified König, methods there was great difficulty in filtering, requiring a long time and giving opportunity for error.

COMMENTS OF REFEREE.

As stated above, the official method for crude fiber does not remove the pentosans, and the König method fails to remove albuminoids, hence the two methods are in no way comparable. The additional treatment with alkali, as in the modified König method, gives us a fiber which is much purer, contains very little nitrogen, and is nearly free from pentosans. The following table is of interest as showing the nitrogen content of the fiber obtained by the three methods:

TABLE V.—*Nitrogen in crude fiber.*

Source.	Official method.	König method.	Modified König method.
	Per cent.	*Per cent.*	*Per cent.*
Distiller's grains	0.27	1.94	0.26
Wheat bran	.00	.195	.00

The results shown in the above table explain why the percentage of fiber obtained by the König method is so much higher than that secured by the official method. This is especially noticeable in the case of the distiller's grains, which carry 5.5 per cent of nitrogen, and your referee believes that the fiber from cottonseed-meal and all feeding materials high in protein, would also carry a high percentage of nitrogen.

In comparing the modified König method with the present official method there are several points to be taken into consideration as mentioned by Messrs. Smith and East in their comments on the König method. Convenience, expense, and, above all, accuracy, are factors which can not be ignored when we are considering any method of chemical analysis. As the modified König method now stands it is not quite so convenient as the official and more expensive, but the point which deserves especial attention is that of accuracy.

Table IV shows that distiller's grains yield nearly 3 per cent and bran 1.25 per cent more fiber when treated as in the official method than when submitted to the action of glycerol-acid mixture with subsequent treatment with alkali.

Your referee does not believe that this difference is due entirely to the presence of pentosans in the fiber from the official method, but that the acid in the presence of glycerol at a temperature of 131° to 133° C. exerts a hydrolytic action upon the fiber in the material. To throw some light upon this point, the effect of the glycerol-acid mixture on pure cellulose was noted. Practically pure cellulose was obtained by treating absorbent cotton, first with dilute sulphuric acid, then with dilute sodium hydrate, washing thoroughly and drying to constant weight in a steam oven at 98° C. The water-free residue was then treated by the König method with results as shown in the following table:

Table VI.—*Treatment of cellulose with glycerol-sulphuric acid.*

No.	Weight of charge (gram).	Amount recovered.	
		Gram.	Per cent.
1	0.2630	0.2305	87.6
2	.2772	.2431	87.7

Of course, if this solvent action takes place in the König method, as the above figures indicate, the modified method would offer no solution to the problem.

Your referee feels some reluctance in condemning the modified method until further evidence is produced corroborating the results shown in the above table. On the other hand, if it be proved conclusively that the acid mixture has no solvent action on the fiber, I believe the modified method can be made practicable by giving special attention to the details of manipulation.

RECOMMENDATIONS.

It is recommended that the referee and associate for 1904 conduct research work mainly along two lines:

(1) An inquiry into the true nature of the ether extract obtained after digesting the residue obtained by the official method of fat extraction, with pepsin.

(2) To prove or disprove that the glycerol-acid mixture of the modified König method has a hydrolytic action upon cellulose.

The **Secretary**. I have a paper on feeding materials from Mr. Browne, of the Louisiana station, which I am requested to submit to the association in his absence.

NOTES ON THE ANALYSIS OF FEEDING MATERIALS.

By C. A. Browne, Jr.

I.—Occlusion of Fat as a Source of Error in Analytical Work.

At the last convention of the Association of Official Agricultural Chemists,[a] the referee on cattle feeds called attention to a statement by Beger regarding the incomplete extraction of fat from certain materials high in protein unless these substances were first subjected to a digestion with pepsin solution. In the course of some feeding experiments recently conducted at the Louisiana Experiment Station upon rice bran and rice polish, an interesting and peculiar confirmation of the above statement was brought to light. With these feeds themselves no appreciative difference was found in the percentage of fat as determined by the ordinary official method and by the pepsin method. On the feces from the steers fed upon these products, however, very decided differences were obtained, the excess of fat by the pepsin method being in one instance over 4 per cent. A comparison of results is given in the following table:

Table I.—*Determination of fat in dried feces by official method and pepsin method.*

Sample of feces.	Time of extraction.	Fat.		Protein.	
		Official method.	Pepsin method.	Before pepsin digestion.	After pepsin digestion.
	Hours.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1. Rice bran	16	9.80	11.56	9.49	4.62
2. Rice polish	16	6.19	6.63	21.50	11.94
3. Rice polish	16	8.17	12.85	23.44	13.56
	24	8.94	....	....	....
	32	10.00	....	....	....

The extraction of the fat by the pepsin method was effected with ease and rapidity, whereas an extraction by the ordinary method, continued for thirty-two hours in one instance, left 3 per cent of fat unremoved. The difficulty was due no doubt to an occlusion of the fat by protein matter. This was further indicated by the fact that in some instances nearly 10 per cent of protein was removed from the dried feces by the pepsin digestion.

The error which may result from an incomplete extraction of fat, either in the food or feces, may seriously vitiate the calculation of digestion coefficients, as is seen from the following table:

Table II.—*Digestive coefficients of fat.*

Sample.	Calculated from results of—	
	Official method.	Pepsin method.
	Per cent.	*Per cent.*
1. Rice bran	59.8	52.0
2. Rice polish	82.5	81.2
3. Rice polish	78.2	66.0

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 73, Proceedings Nineteenth Annual Convention A. O. A. C., p. 148.

Erroneous determinations of fat may thus introduce a considerable error into tables of digestive coefficients, particularly in the case of feeds having a high content of both fat and protein.

A similar source of error in determining fat has been observed in the analysis of mixed feeds containing molasses, the occlusion in this instance being due, not to the protein constituents, but to the dried residue of sugars. With feeds containing molasses, it is recommended to wash 2 to 5 grams of the material on a filter with about 200 cc of cold water, then dry and extract with ether in the usual way.

II.—DETERMINATION OF ACIDITY IN FEEDING MATERIALS.

In the proceedings of the nineteenth annual convention of the association Gudeman describes a method for the determination of acidity in gluten feeds. This method, which consists in titrating an aqueous extract of the feed with standard alkali, gives only the water soluble acid, and is not always satisfactory when the total acidity of a feed is desired, this being particularly the case with feeding materials containing free fatty acids. The following process has been found very satisfactory when the total acidity of a feed is desired:

Twenty grams of the feed are transferred to a 100 cc flask. Eighty cc of 90 per cent alcohol are then added and the flask heated for 30 minutes at a temperature sufficient to keep the contents at a gentle boil. A condensing tube should be used to prevent the loss of alcohol. After cooling, the volume of the flask is made up with 90 per cent alcohol to 105 cc, allowing 5 cc for the volume of feed. After mixing thoroughly the alcoholic solution is poured through a dry filter and 50 cc of the clear filtrate (10 grams of feed) titrated with N/10 alkali. A comparison of the results obtained by direct alcoholic extraction with those calculated from the acid number of the fat in the case of a few samples of rice meal is given herewith:

TABLE III.—*Determination of acidity in feeds.*

Sample.	Acidity by alcoholic extraction.	Acidity calculated from acid number of fat.
	(cc N/10 NaOH per 10 grams feed.)	
1. Rice meal (fresh)	7.6	6.2
2. Rice meal (fresh)	15.5	12.2
3. Rice meal (old sample)	33.7	31.6

The results by alcoholic extraction are a little higher than those calculated from the acid number of the fat, indicating the presence of small amounts of other acid products besides the free fatty acids.

Mr. WILEY. I wish to call attention to a communication received from the international committee on the analysis of foods and feeding stuffs asking for the collaboration of the American chemists. This committee, which includes a member from this country, aims to secure, if possible, uniform methods of analysis for these materials. Two other committees of the same kind have been in operation for several years, namely, the committee on international methods of sugar analysis and a committee on international methods of fertilizer analysis. It is hoped that by this international collaboration the methods employed throughout the world will practically become uniform. They are uniform in essence now but not in detail, and these committees are adopting and considering very carefully, sometimes adopting en bloc, with little or no change, the methods of this association, showing that our work is fully appreciated by these international committees. I happen to be a member of all three of these committees, and so keep in close touch with their work.

On the motion of Mr. Van Slyke the convention adjourned at 2.15 to attend the session of the Association of American Agricultural Colleges and Experiment Stations.

SECOND DAY.

FRIDAY—MORNING SESSION.

The convention was called to order by the president at 9.30 o'clock a. m., when the report of the referee on fats and oils was received.

REPORT ON FATS AND OILS.

By L. M. Tolman, *Associate Referee.*

The Hübl method for determining the iodin absorption of oils and fats is the official method of this association, but it has several faults which affect its accuracy. First, it rapidly loses strength if made up as the official method directs, and in a week or so is unfit for use; second, it is so slow in its reaction with some of the oils, such as linseed, that a serious error is brought about by the change in strength in the solution during the reaction. Wijs[a] shows how great this error might be, and the following table shows how much difference the time of titrating the blank makes in the iodin number.

Table I.—*Iodin number of linseed oil by Hübl method.*

Time of absorption.	Blank titrated at beginning.	Blank titrated at end.
Hours.		
2	173.74	....
7	177.65	170.39
24	181.89	163.16

These figures show a decrease in the iodin number after seven hours, if the blank is titrated at the end of the determination. Wijs considers that the true iodin number in this case lies between 173.7 and 181.89. This change in strength can be greatly lessened by using purified absolute alcohol, but even under these conditions the solution soon becomes so weak that it is of no further use.

Another objection to the Hübl method is that practically each analyst employs a modification of his own, especially, as regards the time necessary for the solution to remain in contact with the oil; and, as is well known, very different results are obtained in this way on the same oils, which results have been collected as data obtained by the Hübl method. The following directions from various authorities as to the time needed for the Hübl test show how varied is the practice in this regard. Allen[b] recommends two hours. Our official method requires three hours.

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[a] Chem. Rev. Fett- u. Hartz-Ind., 1899, **6**: 6.

[b] Com. Org. Anal. **2**: 64, pt. 1. Fett- u. Hartz.

Lewkowitsch[a] recommends four hours. Wijs[b] showed that with linseed oil seven hours was necessary, and it is a very common practice among analysts to allow from 18 to 24 hours. These different practices can not fail to bring about diverse results.

The chief objection to changing the method is that it has been used for a great number of years and a great deal of valuable data on oils and fats has been obtained by its use. If a method can be suggested which will have permanence and speed and still give results which agree with the figures obtained by the Hübl method there seems to be every reason for discarding the old method.

Three solutions have been suggested as substitutes for the Hübl method which overcome to a great degree its faults, and a study of these methods has been the work of the referee this year. Cooperative work with chemists on the Wijs and Hanus methods has been done, while the bromin solution has been a subject of special study by the referee, as has also the excess of reagents necessary and the speed of reaction of the other solutions.

The keeping qualities of the Wijs and Hanus solutions have been thoroughly tested and found to be very satisfactory when the acetic acid is pure and of 99 per cent strength. The bromin solution has not been so thoroughly tested, but a solution of N/3 bromin in pure carbon tetrachlorid showed very little change in strength in 5 months, which would make it perfectly satisfactory on that score. Some peculiarities of the bromin solution will be noted later.

The cooperative work was limited to a comparison of the results obtained on various oils by the Hübl, Hanus, and Wijs methods, the object being to find what difference in the present limits on iodin numbers would be brought about by adopting either of the new methods. Six samples of oils, with the following directions, were sent out by the referee to a number of chemists interested, with the request that careful comparative work be done on these with both the Hanus and Wijs, and that figures be reported on such other fats and oils as might be available.

DIRECTIONS FOR IODIN WORK.

Hübl method.—Follow method as given in Bulletin No. 46 or No. 65. Allow excess of at least 50 per cent of the total iodin added.

Hanus method.—Dissolve 13.2 grams iodin in 1,000 cc glacial acetic acid (showing no reduction with bichromate and sulphuric acid); add enough bromin to double the halogen content determined by titration. Three cubic centimeters of bromin is about the proper amount. The iodin may be dissolved by the aid of heat, but the solution should be cold when bromin is added.

Wijs method.—Dissolve 13.2 grams iodin in acetic acid, same as used in Hanus solution, and run in current of dried and purified chlorin gas until the halogen content is nearly doubled; a slight excess of iodin should be allowed. The solution may also be made by dissolving 10 grams iodin trichlorid in glacial acetic acid and adding 10.8 grams of iodin.

These two solutions are used in the same manner as in the Hübl method except that an excess of 70 per cent of the iodin added is allowed; 10 cc of the potassium iodid solution is added instead of 20 cc, and the time of reaction is shortened to 15 minutes and 30 minutes, depending on whether a nondrying oil, a drying oil or a fat is tested. After the potassium iodid solution is added the mixture must be shaken thoroughly before adding the water.

In reporting results state details as to methods and manipulation.

References:
Hanus: Zeits. Nahr. Genussm., 1901, 4: 913; J. Soc. Chem. Ind., 1902, p. 455.
Wijs: Berichte, 1898, p. 752; J. Soc. Chem. Ind., 1902, p. 455.

As a result of the remarkable interest shown, enough figures have been reported on many kinds of fats and oils to give a very satisfactory basis of comparison for the three methods. These results have been collected in the following tables. Table II was prepared from results received on the samples sent out by the associate referee.

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[a] Chem. Analysis of Oils, Fats, and Waxes, 1899, p. 173.

[b] Chem. Rev. Fett- u. Hartz-Ind., 1899, 6: 7.

TABLE II.—*Results on official samples of oils.*

Kind of oil.	Hübl number.	Hanus number (30 minutes).	Wijs number (30 minutes).	Difference Hanus and Hübl numbers.	Difference Wijs and Hübl numbers.	Analyst.
Olive	89.8	90.0	91.4	+ 0.2	+ 1.6	Tolman and Munson.
Do	89.0	89.6	90.5	+ .6	+ 1.5	Blasdale.
Do	88.3	90.2	92.2	+ 1.9	+ 3.9	Van Wormer.
Do	87.0	88.2	90.8	+ 1.2	+ 3.8	Adams.
Do	89.5	90.2	90.2	+ .7	+ .7	Lane.
Rape	101.3	105.2	105.7	+ 3.9	+ 4.4	Tolman and Munson.
Do	100.6	103.2	104.7	+ 2.6	+ 4.1	Blasdale.
Do	99.9	104.3	103.1	+ 4.4	+ 3.2	Van Wormer.
Do	102.0	101.8	105.2	— .2	+ 3.2	Adams.
Do	101.3	101.5	105.7	+ .2	+ 4.4	Lane.
Mustard	113.6	116.8	118.2	+ 3.8	+ 5.2	Tolman and Munson.
Do	111.7	115.2	116.9	+ 3.5	+ 5.2	Blasdale.
Do	114.1	116.2	118.4	+ 2.1	+ 4.3	Van Wormer.
Do	112.4	113.5	117.9	+ 1.1	+ 5.5	Adams.
Do	112.4	113.0	116.9	+ .6	+ 4.5	Lane.
Maize	123.3	126.0	129.2	+ 2.7	+ 5.9	Tolman and Munson.
Do	114.1	123.4	125.9	+ 9.3	+11.8	Blasdale.
Do	118.8	123.1	126.8	+ 4.3	+ 8.0	Van Wormer.
Do	120.9	122.0	124.8	+ 1.1	+ 3.9	Adams.
Do	121.9	121.7	125.7	— .2	+ 3.8	Lane.
Poppy	134.9	138.4	139.1	+ 3.5	+ 4.2	Tolman and Munson.
Do	133.9	135.1	136.7	+ 1.2	+ 2.8	Blasdale.
Do	135.1	134.9	134.9	— .2	— .2	Van Wormer.
Do	132.8	132.4	136.0	— .4	+ 3.2	Adams.
Do	138.2	136.4	138.5	— 1.8	— .3	Woodman.
Do	132.0	133.3	135.8	+ 1.8	+ 3.8	Lane.
Linseed	....	183.7	188.7	....	....	Tolman and Munson.
Do	167.7	185.1	188.4	+17.4	+20.7	Blasdale.
Do	166.7	186.1	188.6	+19.4	+21.9	Van Wormer.
Do	173.0	180.9	187.2	+ 7.9	+14.2	Adams.
Do	165.1	186.3	189.2	+21.2	+24.1	Lane.

All these oils have a comparatively high iodin number and the agreement between the results obtained by the different methods is not so close as is found with the fats and oils of a low iodin number. The Hübl method, especially on the oils with high iodin numbers, shows many discrepancies between the results of the various analysts. The Wijs method gave the best results; especially is this true with linseed oil where the maximum difference between the results of the five analysts was 2.00 numbers. The Hanus method also gives good results, which are uniformly lower than those of the Wijs method, making a closer agreement with figures at present obtained by the Hübl method. This agreement, however, is not of great value on these oils, especially the rape, mustard, maize, poppy, and linseed oils, as the published data give such wide limits.

These wide limits are doubtless largely due to the varying methods and modifications of the Hübl method used in the determination. The Hübl solutions used in this work were not more than 24 hours old, and the results show this wide variation. If they had varied in age from one week to three weeks, probably a very much wider variation would have been found. This point is illustrated in the table following, taken from Benedikt and Ulzer, page 205.

Table III.—*Results obtained with Hübl solutions of varying ages.*

Age of Hübl solution.	Excess of iodin.	Iodin number of linseed oil.	Equivalent of 25 cc of solution in N/10 thio.
Days.	*Per cent.*		*Cc.*
1	62.5	201.0	47.11
2	64.0	198.5	45.7
4	65.0	196.7	44.25
11	67.0	191.0	38.89
26	68.0	185.9	31.81

These figures show a difference of 5 numbers with the same solution. It is on oils of this kind that the new solutions will give much more satisfactory results.

Table III contains the results obtained by G. E. Patrick on 51 butters and 14 oleomargarins by the Hübl and Hanus methods. In every case he obtained slightly higher results with the Hanus method than with the Hübl. The average on 51 samples of butter was 1.01 numbers, and the maximum was 1.97 numbers. On the 14 samples of oleomargarin the average was 1.14 numbers higher and maximum of 1.61 numbers. These results were on fresh and rancid products, the results showing no difference.

Table IV.—*Comparison of Hübl and Hanus methods on butter and oleomargarin* (*G. E. Patrick*).

BUTTERS.

Laboratory No.	Hübl.		Hanus.		Difference between Hanus and Hübl numbers.	Remarks.
	No.	Time.	No.	Time.		
		Hrs.		*Min.*		
139	33.68	4	34.59	30	+0.91	
140	28.98	4	29.42	75	+ .44	
141	31.39	4	32.00	60	+ .61	
142	30.20	4	30.47	30	+ .27	
143	30.57	4	30.82	60	+ .25	
144	36.24	4	37.06	60	+ .82	
145	35.27	4	36.38	30	+1.11	
146	34.50	4	35.62	30	+1.12	
147	32.16	4	33.15	30	+ .99	
927	29.24	4	29.84	30	+ .64	
928	35.97	4	36.56	30	+ .58	
151	29.51	3	31.25	30	+1.74	
153	35.62	3	37.59	30	+1.97	
27	28.11	3	29.99	30	+1.88	Very rancid.
35	31.59	3	33.28	30	+1.69	
32	33.38	3	34.67	30	+1.29	
75	26.90	3	27.96	30	+1.06	
78	36.79	3	38.28	30	+1.49	
864	35.04	3	36.95	30	+1.91	
871	29.48	3	31.24	30	+1.76	
872	31.41	3	32.71	30	+1.30	
1089	43.08	4	44.59	30	+1.51	Fresh.
1102	31.38	4	32.24	30	+ .86	
1108	33.66	4	34.14	30	+ .48	

TABLE IV.—*Comparison of Hübl and Hanus methods on butter and oleomargarin* (*G. E. Patrick*)—Continued.

BUTTERS—Continued.

Laboratory No.	Hübl.		Hanus.		Difference between Hanus and Hübl numbers.	Remarks.
	No.	Time.	No.	Time.		
		Hrs.		*Min.*		
73	34.19	4	34.76	30	+ .57	Very rancid.
82	35.52	4	36.23	30	+ .71	
85	35.53	4	36.38	30	+ .85	
....	41.05	4	42.35	30	+1.30	
1141	39.75	4	41.27	30	+1.52	
1089	42.86	4	44.27	30	+1.41	
1090	43.03	4	44.44	30	+1.41	
81	39.65	3¼	40.67	30	+1.02	Rancid.
84	34.45	3¼	34.97	30	+ .52	
871	27.85	3¼	28.31	30	+ .46	
872	30.24	3¼	30.58	30	+ .34	
151	28.54	3¼	29.03	30	+ .49	
153	33.93	3¼	34.69	30	+ .76	
74	34.98	4	36.12	30	+1.14	Do.
76	35.76	4	36.77	30	+1.01	
77	36.98	4	38.28	30	+1.30	
79	35.90	4	36.77	30	+ .87	
80	39.48	4	40.92	30	+1.44	
83	32.70	4	33.66	30	+ .96	
1157	39.77	4	40.04	30	+ .27	Fresh.
1159	38.73	4	39.64	30	+ .91	
1160	41.30	4	42.54	30	+1.24	
1161	37.28	4	37.94	30	+ .66	
1162	35.69	4	36.65	30	+ .96	
1163	32.52	4	33.43	30	+ .91	
1156	37.72	4	38.52	30	+ .80	
1158	39.52	4	40.27	30	+ .75	
					+1.01	Average.
					+1.97	Maximum.
OLEOMARGARIN.						
49	46.38	3	47.99	60	+1.61	Very rancid.
37	41.58	3	43.12	45	+1.54	Do.
XXX	49.39	3	50.69	30	+1.30	Do.
118	59.93	3	61.01	30	+1.08	Do.
1010	54.74	3	55.70	30	+ .96	Do.
21805	46.45	3	47.62	30	+1.17	Two years old.
21920	66.17	3	66.65	30	+ .48	
21923	65.29	3	66.44	30	+1.15	
21925	52.94	3	53.84	30	+ .90	
22287	68.91	3	69.18	30	+ .27	
22548	50.68	3	52.06	30	+1.38	
23375	46.64	3	48.05	30	+1.41	
995	72.39	3	73.90	30	+1.51	Four months old.
1073	72.24	3	73.51	30	+1.27	Three months old.
					+1.14	Average.
					+1.61	Maximum.

TABLE V.—*Results on miscellaneous oils and fats.*

Kind of oil or fat.	Hübl number (3 hours).	Hanus number (30 minutes).	Wijs number (30 minutes).	Difference between Hanus and Hübl numbers.	Difference between Wijs and Hübl numbers.	Analyst.
Cocoanut........	8.93	8.60	9.05	− 0.33	+ 0.12	Tolman and Munson.
Do...........	8.31	8.12	....	− .19	....	Blasdale.
Butter...........	35.3	35.3	36.2	.00	+ .90	Tolman and Munson.
Do...........	34.8	35.4	35.9	+ .60	+ 1.10	Do.
Do...........	28.96	29.91	30.48	+ .95	+ 1.52	Woodman.
Do...........	29.90	30.0	30.60	+ .10	+ .70	Van Wormer.
Do...........	35.5	35.4	....	− .10	....	Adams.
Do...........	41.0	41.4	....	+ .40	....	Do.
Do...........	42.1	41.9	....	− .20	....	Do.
Do...........	40.5	41.0	....	+ .50	....	Do.
Oleomargarin ...	42.6	43.3	43.5	+ .70	+ .90	Tolman and Munson.
Do...........	53.6	52.3	53.5	− 1.30	− .10	Do.
Do...........	52.8	52.2	53.7	− .60	+ .90	Do.
Do...........	52.5	52.0	52.9	− .50	+ .40	Do.
Do...........	66.3	64.8	66.0	− .30	− 1.50	Do.
Do...........	64.33	65.7	66.4	+ 1.40	+ 2.10	Woodman.
Do...........	52.00	52.0	52.2	.00	+ .20	Van Wormer.
Do...........	66.1	67.8	67.5	+ 1.70	+ 1.40	Do.
Do...........	47.7	48.5	....	+ .80	....	Adams.
Do...........	46.6	47.6	....	+ 1.00	....	Do.
Do...........	49.8	50.5	....	+ .70	....	Do.
Do...........	46.4	48.0	....	+ 1.60	....	Do.
Do...........	50.2	51.8	....	+ 1.60	....	Do.
Do...........	48.8	48.9	....	+ .10	....	Do.
Do...........	47.3	48.2	....	+ .90	....	Do.
Olive	82.55	84.6	....	+ 2.1	....	Woodman.
Cotton seed......	103.8	105.2	105.3	+ 1.40	+ 1.50	Tolman and Munson.
Do...........	106.2	107.8	107.3	+ 1.60	+ 1.10	Do.
Do...........	104.8	106.7	106.2	+ 1.90	+ 1.40	Do.
Do...........	111.90	111.2	113.4	− .70	+ 1.50	Woodman.
Do...........	95.84	96.5	99.9	+ .66	+ 4.1	Van Wormer.
Do...........	94.09	97.47	96.97	+ 3.38	+ 2.88	Blasdale.
Do...........	108.2	106.6	....	− 1.6	....	Adams.
Do...........	106.6	104.7	....	− 1.9	....	Do.
Do...........	105.6	103.1	....	− 2.5	....	Do.
Peanut	96.3	97.4	99.0	+ 1.10	+ 2.70	Tolman and Munson.
Do...........	94.5	94.1	95.2	− .40	+ .70	Do.
Do...........	107.7	107.7	109.5	.00	+ 1.80	Do.
Mustard	110.4	115.5	118.5	+ 5.10	+ 8.10	Do.
Do...........	113.0	116.8	118.2	+ 3.80	+ 5.20	Do.
Do...........	98.4	103.8	104.3	+ 5.40	+ 5.90	Do.
Do...........	103.5	110.2	112.5	+ 6.70	+ 9.00	Do.
Do...........	106.4	114.8	117.3	+ 8.40	+10.90	Do.
Rape	101.3	105.2	105.7	+ 3.90	+ 4.40	Do.
Do...........	100.2	102.8	104.1	+ 2.60	+ 3.90	Do.
Sunflower	106.4	107.2	109.2	+ .80	+ 2.80	Do.
Sesame	106.4	106.5	107.0	+ .10	+ .60	Do.
Do...........	95.9	103.5	100.8	+ 7.6	+ 4.9	Blasdale.
Maize	119.0	120.2	123.2	+ 1.20	+ 4.20	Tolman and Munson.
Do...........	119.0	119.6	122.2	+ .60	+ 3.20	Do.
Poppy	133.4	132.9	135.2	− .50	+ 1.80	Do.
Linseed	169.8	184.5	186.5	+14.70	+16.70	Do.

Mr. Patrick makes the following comment:

> The Hanus solution has two strong points in its favor, namely, good keeping qualities and rapidity of action on the fats. The latter feature is important not alone because it expedites work, but also because it very largely obviates the errors due to expansion of liquids, provided blanks be run at rather frequent intervals throughout the day's work. The advantages of the Hanus solution are so marked that I have adopted it for regular use.

Mr. Patrick's results and those reported by the other analysts on butter and oleomargarin indicate that the Hanus method could be substituted for the Hübl in the examination of butter and similar products without any appreciable change in the limits at present held on these products. The same may also be said of the Wijs reagent, although in nearly every case the Wijs shows slightly higher results on these fats than the Hanus.

Table V contains the results on miscellaneous oils and fats sent in by the various chemists.

Table VI contains the results on 11 samples of pure olive oil prepared by G. E. Colby, of the University of California, and sent by him to W. C. Blasdale and the referee. These results show that the Hanus solution gave the best comparative results, there being a smaller average difference among results obtained by that method than with others, and a lower maximum difference, the Hübl method showing the greatest difference and the Wijs the next. This would indicate that the Hanus and Wijs methods had a tendency to give more uniform results than the Hübl, which is certainly a strong point in their favor. This fact was also noted in Table II giving results on the official samples. It is also evident from Table VI that the Wijs solution gives slightly higher results on olive oils. This confirms the referee's previous work[a] in which the average difference on 36 samples was +1.2 numbers.

TABLE VI.—*Pure olive oils.*

Number.	Hübl number.			Hanus number.			Wijs number.			Maximum difference.		
	Tolman.	Blasdale.	Colby.	Tolman.	Blasdale.	Colby.	Tolman.	Blasdale.	Colby.	Hübl.	Hanus.	Wijs.
1	83.7	82.44	83.1	83.7	83.60	83.7	85.0	83.44	83.7	1.3	0.1	1.6
2	83.7	81.69	83.7	84.0	83.24	83.0	85.9	83.73	84.2	2.0	1.0	2.2
3	79.9	77.70	79.7	79.1	79.51	80.5	81.3	79.22	81.3	2.2	1.4	2.1
4	86.1	85.40	85.0	85.6	86.65	85.5	86.6	85.57	87.9	1.1	1.1	2.4
5	83.2	83.53	82.9	83.0	84.24	83.9	84.4	83.13	83.7	.6	1.2	1.3
6	84.6	82.90	83.8	84.6	85.64	85.5	85.4	83.84	86.0	1.7	1.0	2.2
7	90.6	87.37	90.0	89.8	89.46	90.5	91.5	90.62	91.4	3.3	1.1	.9
8	85.4	83.42	84.8	85.1	84.88	86.7	86.7	85.03	85.3	2.0	1.9	1.7
9	84.8	82.69	85.1	84.1	84.18	85.0	85.9	84.16	86.9	2.4	.9	2.8
10	86.1	84.47	85.8	86.7	85.75	87.6	87.8	86.65	87.0	1.7	1.9	1.2
11	84.4	84.72	83.0	84.1	85.49	84.0	85.1	84.66	84.6	1.7	1.4	.5
Average	......	....	......	......	....	......	......	....	......	1.8	1.2	1.7
Maximum	......	....	......	......	....	......	......	....	......	3.3	1.9	2.8

The Hanus figures, obtained by the different analysts, do not have quite the same close agreement. The referee has obtained very close agreement between Hanus and Hübl. On 36 samples previously reported the average difference was 0.59, and on the 11 samples of Table VI an average of 0.24. Blasdale gets a slightly wider difference of 1.4 numbers, while Colby gets 0.89. The average of all would give less than 1. These results seem to show that these solutions are satisfactory substitutes.

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[a] Tolman and Munson, J. Amer. Chem. Soc., 1903, **25**: 244.

TABLE VII.—*Cooperative work by N. J. Lane.*

Substance.	Iodin values of straight oil.								Iodin values of liquid acids of oils.						Remarks.
	Hübl method.		Hanus method.			Wijs method.			Per cent of liquid acid.	Hübl method 14 hours.	Hanus method.		Wijs method.		
	3 hours.	6 hours.	15 minutes.	30 minutes.	60 minutes.	15 minutes.	30 minutes.	60 minutes.					1 hour.	2 hours.	
Cotton-seed oil	104.9	......	......	104.5	106.4	......	107.4	109.2	75.58	136.3	1 hour. 139.7	......	......	......	"Off" oil.
Do	108.2	......	......	108.1	108.8	......	112.5	112.2	77.18	139.0	1 hour. 135.3	3 hrs. 15 min. 152.4	......	......	"White" oil.
Do	111.3	......	......	109.2	110.0	......	113.7	111.6	78.79	140.2	1 hour. 140.7	2 hours. 147.1	145.1	143.7	White oil Hübl value is average of several determinations.
Steam lard	65.97	......	65.94	65.19	......	72.11	72.93	......	60.64	91.24	1 hr. 20 min. 91.54	2 hours. 89.71	98.83	101.7	Liq. acids by petroleum ether; per cent too low.
Tallow	51.74	......	53.68	52.84	......	55.27	53.89	......	58.79	85.39	1 hour. 84.28	2 hrs. 15 min. 84.74	......	......	Hanus 3 hrs. 10 min.=86.98.
Linseed oil	165.1	181.4	......	186.3	185.7	......	189.2	185.9	......	......	......	......	......	......	A. O. A. C. sample.
Olive oil	89.50	......	90.22	90.16	......	90.10	90.20	......	......	......	......	......	......	......	A. O. A. C. sample Aug. 25, 1903.
Maize oil	121.9	......	......	121.7	123.2	......	125.7	125.4	......	......	......	......	......	......	A. O. A. C. sample Aug. 27, 1903.
Mustard-seed oil	112.4	......	......	112.4	113.0	......	116.9	116.3	......	......	......	......	......	......	A. O. A. C. sample Aug. 28, 1903.
Poppy oil	132.0	......	......	133.3	134.9	......	135.8	136.8	......	......	......	......	......	......	A. O. A. C. sample Aug. 28, 1903.
Rape	101.3	......	......	101.5	101.5	......	105.8	105.6	......	......	......	......	......	......	A. O. A. C. sample Oct. 17, 1903.
Cotton-seed oil and tallow	......	......	86.06	......	......	......	......	......	68.42	118.0	1 hour. 126.5	......	......	......	
Do	......	......	60.27	......	......	......	......	......	53.53	103.9	1 hour. 105.2	......	......	......	

Table VII contains the results reported by N. J. Lane, and includes some results on the iodin numbers of the liquid fatty acids of the various oils and fats. He makes the following comment in regard to the merits of the methods:

> I think the Hanus is so far ahead that it requires no comment as to its use on the straight oils. The differences between it and the Hübl are so slight as to be negligible. Different samples of the same oil vary more than the results by these two methods on the same sample, and therefore it could readily be substituted for the Hübl without any inconvenience or change of standards.
>
> Regarding the action of the Hanus solution on the liquid acids, the results do not agree very well, either among themselves or with the Hübl, but this may be a matter of time of action. For some reason liquid acids require more time for complete absorption of iodin than the straight oil and it is useless to attempt to get the Hübl value in less than 12 to 14 hours. The difference between the Hübl and the Wijs methods seems to render the Wijs impracticable for general use without a revision of all standards. It does not possess any advantage over the Hanus, and the solution is far more troublesome to prepare.

COMMENTS BY COOPERATING ANALYSTS.

W. C. Blasdale.—As to the relative merits of the three processes I think I should, on the whole, prefer the Hanus. The manipulation is quite as satisfactory as the Hübl and the shorter time required for a determination is a very decided advantage as well as the greater permanence of the solution.

A. G. Woodman.—I think, on the whole, I prefer the Hanus method to the Wijs for convenience in preparation of the solution and in use. Either is far preferable to the Hübl. I suggest the following methods for dissolving the iodin and weighing the fat:

HANUS SOLUTION.

> Dissolve C. P. iodin in glacial acetic acid. Place the iodin in a flask immersed in hot water and add the acid in small amounts with frequent shaking and decanting until solution is complete. To the cold solution add the required amount of bromin.

WEIGHING SAMPLES.

> Weigh out the oil in a small beaker containing also a short piece of glass tubing drawn out like a burette tip. To the upper end of this attach a small bit of rubber tubing containing a glass bead to close the end. Weigh the whole system, then by compressing and releasing the rubber tube draw a quantity of oil partly up the glass tube, transfer the required number of drops to the bottle and weigh the system again. Weigh the solid fats at such a temperature that they are liquid and homogeneous. Calculate the amount taken in each case so that about 70 per cent of the iodin is in excess.

A. B. Adams.—In their workings the Hanus and the Wijs methods are very similar, but of the two we prefer the Hanus, for the following reasons:

> 1. In making up the solutions, bromin is more easily obtained than iodin trichlorid.
> 2. The figures obtained in the Hanus are very nearly the same as those given by the official method (Hübl).

Of the three, Hanus, Wijs, and Hübl, we prefer the Hanus, for the following reasons:

> 1. Length of time required to make up the reagent—Hanus being ready immediately, the Hübl not until it has stood 12 hours.
> 2. The Hübl solution weakens rapidly, the Hanus very slowly.
> 3. With the Hanus method nearly all oils require only about 30 minutes for complete absorption, while with the Hübl 3 hours is not sufficient.
> 4. In agreement of duplicates the Hanus method is far superior; in fact, there is no comparison between the two.

There seems to be every reason in favor of the Hanus method. In our work the oleo is allowed to stand 30 minutes, as we think this gives safer figures in all cases.

Linseed oil should be allowed to stand 1 hour. In short, 30 minutes for oils or fats with a figure below 150, with a figure over that, 1 hour should be long enough.

R. E. Doolittle.—The only difficulty experienced with these methods was the weighing out of the trichlorid. This could be obtained in 1-oz. bottles, or even ½-oz., and the whole bottle used, which would do away with the difficulty.

The remainder of the paper is a report of work done by the referee in the Bureau of Chemistry:

THE WIJS SOLUTION.

Wijs[a] directs that there be an excess of 70 per cent of the iodin added in the determination in order to insure complete and rapid absorption, but the results on linseed oil given in Table VIII indicate that such a large excess is unnecessary. In each of the determinations 0.1500 gram of linseed oil was used and the solution was allowed to stand 30 minutes in contact with oil.

TABLE VIII.—*Iodin absorption of linseed oil with varying excess.*

Wijs solution.	Value in N/10 iodin.	Amount of iodin absorbed in N/10 iodin.	Iodin number.	Excess of iodin.
cc	*cc*	*cc*		*Per cent.*
25	61.28	23.16	193.4	62
20	49.35	23.13	193.7	53
15	37.08	23.13	193.7	37

These figures show that with an excess of only 37 per cent the same results were obtained in 30 minutes as with an excess of 62 per cent. Linseed oil was used on account of its high iodin number and because it takes longer to obtain complete absorption with it than with oils of a lower iodin value. This smaller excess effects a great saving in solution and makes titrations more rapid. This smaller excess does not greatly affect the speed of reaction, as is shown in Table IX.

TABLE IX.—*Effect of excess of Wijs solution on speed of reaction.*

Time.	Iodin numbers.	
	Excess of iodin 62 per cent.	Excess of iodin 37 per cent.
Minutes.		
1	191.1	189.0
5	191.9	191.2
10	191.1	191.4
15	191.7	191.4
30	192.3	191.4
60	193.1	....

These results show that the reaction between the Wijs reagent and the oil is very rapid, even with only 37 per cent excess.

Table X shows that when the determinations are made in the light the iodin number seems to increase up to a definite point where it remains constant for a short time, then gradually increases up to 4 hours with no tendency to remain constant. In the dark the iodin number remained constant from 1 to 2 hours.

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[a] Zeit. Nahr. Genussm., 1902, 5: 499.

TABLE X.—*Iodin number of linseed oil by Wijs solution.*

Time.	Iodin numbers.	
	In light.	In dark.
Minutes.		
1	192.4	186.1
10	193.1	192.6
30	193.1	192.9
60	194.1	193.5
120	194.7	193.4
240	195.7	....

TABLE XI.—*Effect of time and light on Wijs and Hanus solutions.*

Time.	Iodin numbers.	
	In light.	In dark.
WIJS.		
30 minutes.	191.9	192.1
5 hours	194.0	193.0
HANUS.		
30 minutes.	189.1	189.2
5 hours	191.9	192.1

It will be noticed from these results that on the Wijs solution, standing 30 minutes, the light has no effect; the same may be said of the Hanus. In the 5-hour test two effects are noted in the Wijs, namely, there is a very slight increase in the iodin number over the figures obtained in 30 minutes, and the results obtained in the light are slightly greater than those obtained in the dark. With the Hanus solution there is a slight increase in 5 hours over 30 minutes, but no difference in light or dark. This would indicate that there it is not necessary to stand either the Wijs or Hanus solution in a dark cupboard when 30 minutes are allowed for the reaction.

This increase of absorption on long standing is probably due to secondary reactions as is shown in the results with iodin chlorid in carbon tetrachlorid given in Table XI. Upon standing longer than thirty minutes substitution takes place and longer standing shows a remarkable, though gradual, change in the results.

TABLE XI.—*Iodin number by iodin chlorid in carbon tetrachlorid.*

Time.	Total.	Addition.	Substitution.
Hours.			
5	210.8	184.9	12.9 in light.
5	204.8	190.9	6.9 in dark.
1	198.6	194.2	2.2 in light.
½	195.2	194.6	.80 in light.

These figures show that the addition apparently decreases with time after 1 hour, with a gradual increase in the total amount of iodin entering into combination. This indicates that with iodin chlorid in carbon tetrachlorid, standing longer than 30 minutes is no advantage and longer than 1 hour gives entirely unreliable figures. It seems probable that the same thing will be shown with iodin chlorid in acetic acid although to a much less extent, and that long standing gives untrue results. With iodin bromid in carbon tetrachlorid the secondary reactions are much weaker, but the gradual increase up to 18 hours' standing with Hanus solution is doubtless due to the same cause. This indicates that there is a maximum as well as a minimum time which should be allowed for the reaction, and for the Wijs solution this limit is between 15 minutes and 1 hour, the advantage being with the shorter time rather than the longer, 30 minutes being satisfactory for either solution.

THE HANUS SOLUTION.

Hanus[a] also advises an excess of 70 per cent in order to insure complete absorption, and the results given in Table XII show that nearly this large excess is necessary to obtain complete absorption on linseed oil in 30 minutes.

TABLE XII.—*Iodin number of linseed oil with varying excess of iodin.*

Wijs solution.	Value in N/10 iodin.	Amount of iodin absorbed in N/10 iodin.	Iodin number.	Excess of iodin.
cc	*cc*	*cc*		*Per cent.*
25	54.76	23.26	196.3	57
20	44.08	22.73	191.9	48
15	33.06	21.86	184.5	34

These figures, if compared with Table VIII, show how much slower in reaction the Hanus solution is than the Wijs, and how much greater excess is needed to produce the same results. Table XIII also brings out the slowness of the Hanus solution as compared with the Wijs.

TABLE XIII.—*Iodin numbers by Hanus solution in varying times.*

Time.	Iodin No. 1. In light.	Iodin No. 2. In light.	Iodin No. 3. In dark.	Iodin No. 4. In dark.	Iodin No. 4. In light.
Minutes.					
1	164.8	164.6	166.9	....	....
5	178.9	....	....	....	....
10	181.3	182.2	187.7	....	....
15	184.6	186.7	....	....	....
30	185.6	187.5	191.0	189.2	189.1
Hours.					
1	185.6	187.5	191.3	....	....
2	....	188.5	191.0	....	....
4	....	189.5	....	....	....
5	....	....	....	192.1	191.9
18	....	193.3	....	....	....

These results on samples of linseed oil by different analysts show that it takes 30 minutes by the Hanus solution to reach the constant stage which is reached by the Wijs in 10 minutes. The absorption is constant from 30 minutes to 1 hour and then begins the gradual rise, as was the case with the Wijs, and the same explanation is suggested. The Hanus is less affected by light than the other solutions.

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[a] Zeit. Nahr. Genussm., 1901, 4: 913.

BROMIN SOLUTION AND CARBON TETRACHLORID AS A SOLVENT IN THE WIJS AND HANUS METHODS.

In connection with the work of testing the Hanus and Wijs solutions, it was thought well to test the bromin solution proposed by McIlhiney and also the substitution of carbon tetrachlorid as a solvent for acetic acid in the Hanus and Wijs methods. The advantage of this solvent is that it is neutral and permits of an estimation of the substitution which takes place in the reaction between the oil and the halogen. It was thought that if a change was to be made in our present method and any information of added value could be obtained by the use of a solvent of this nature, it would be well to adopt at this time the best method obtainable.

Solutions of iodin chlorid, iodin bromid, and bromin in carbon tetrachlorid were prepared, using the same strength as in the Wijs and Hanus methods and approximately N/3 bromin as directed by McIlhiney. A solution in carbon tetrachlorid of linseed oil of known purity was prepared and used in most of the experiments. The actual amount of oil used in each test was affected to a very slight degree by changes in temperature. As a rule, a fresh solution was made up for each set of tests and only very slight variations were found in the iodin numbers obtained by the Wijs and Hanus solutions in acetic acid at any time. In each determination 0.1500 of a gram of oil was used, which made it possible to regulate the excess of reagent and other conditions very exactly.

With these solutions several questions were to be decided:

1. How do the results obtained compare with those given by other solutions, especially the Hübl?
2. How long a time is required to obtain maximum figures?
3. Does water affect the amount of substitution?
4. How does light affect the reaction between oil and the halogen?
5. Does age of solution affect the results?

At this point it might be well to consider what results would be obtained if the acid formed in the Hübl reaction is estimated as substitution and the addition figure calculated from these figures. The formation of acid in the Hübl reaction has long been known and many suggestions have been made as to its cause; but the reaction has not as yet been satisfactorily explained, although it has been proved that it is not due to substitution.[a]

In Table XIV are collected the results on a linseed oil by the Hübl method, the acid formed being determined by addition of potassium iodate.

TABLE XIV.—*Results on linseed oil by the Hübl method, with addition of potassium iodate.*

Time of reaction.	Hübl number.	Addition.	Substitution.
Hours.			
½	161.9	124.7	18.6
1	165.0	127.4	18.8
2	170.5	130.9	19.8
3	175.1	135.5	19.8
5	180.0	146.4	16.8
24	181.0	153.2	13.9

The amount of acid remains practically the same, regardless of the time of reaction, the most satisfactory explanation being that there is a decomposition of the addition product formed in the reaction and the formation of an acid due largely to the presence of water. The occurrence of this decomposition can be easily proved by adding potassium iodate after titrating the excess of free iodin and noting the rapid formation of free iodin on standing, which takes place so quickly that the determination of substitution or acid must be made with great rapidity or very different results will be obtained. These results are given as being suggestive in connection with those to be discussed later. The bromin solution will be considered first, the figures being given in Tables XV and XVI:

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[a] Ingle, J. Soc. Chem. Ind., 1902, p. 587.

TABLE XV.—*Iodin numbers obtained by three different methods.*

Time.	Wijs method.				Hanus method.				Bromin method (in light).		
	Acetic acid.	Carbon tetrachlorid.			Acetic acid.	Carbon tetrachlorid.			Total.	Addition.	Substitution.
		Total.	Addition.	Substitution.		Total.	Addition.	Substitution.			
Minutes.											
1	186.1	......	......	......	166.9	164.0	164.0	0.0	156.3	156.3	0.0
2	......	192.8	192.8	0.0	......	......	......	......	......	......	......
10	192.6	193.6	193.6	0.0	187.7	182.5	182.5	0.0	......	......	......
15	......	......	......	......	......	......	......	......	......	......	......
30	192.9	195.2	194.6	.80	191.1	188.6	188.6	0.0	193.2	193.2	0.0
60	193.5	198.6	194.2	2.20	191.3	189.7	189.7	0.0	197.0	197.0	0.0
120	193.4	......	......	......	191.0	......	......	......	196.7	194.2	1.27

TABLE XVI.—*Iodin numbers obtained by varying time of reaction in carbon tetrachlorid solution.*

Solution.	1 minute.			10 minutes.			30 minutes.			60 minutes.			120 minutes.		
	Total.	Addition.	Substitution.	Total.	Addition.	Substitution.	Total.	Addition.	Substitution.	Total.	Addition.	Substitution.	Total.	Addition.	Substitution.
Bromin	156.0	156.0	0.0	......	......	......	193.2	193.2	0.0	197.4	194.4	0.0	196.6	194.2	1.27
Do.	156.4	156.3	0.0	186.3	186.3	0.0	194.0	194.0	0.0	198.2	198.0	0.0	203.8	202.0	.93
ICl in CCl_4	192.8	192.8	0.0	193.6	193.6	0.0	196.2	194.6	0.80	198.6	194.2	2.2	......	......	......
IBr in CCl_4	164.2	164.2	0.0	182.5	182.5	0.0	188.6	188.6	0.0	189.7	189.7	0.0	......	......	......

Table XV gives a comparison of the values obtained by the different methods on a sample of oil giving a Hübl number of 180. The solutions were all freshly prepared and C. P. carbon tetrachlorid, perfectly dry, was used in making them up. The results obtained show a very satisfactory agreement. The substitution figures are very small in all the solutions and appeared only after long standing in the Wijs and bromin solutions, being largest with the Wijs. The maximum addition figure was not reached with any of the solutions in less than one hour, as is shown in Table XVI. The results with the bromin solution were so different from those obtained on the same oil with other bromin solutions that the cause was sought with considerable interest. The relative speed of these solutions is about the same as with acetic acid as the solvent, as might be expected. The Wijs reagent is by far the most active, as shown in the other cases.

First a little water was added to the solution of oil in carbon tetrachlorid; then the fresh bromin solution was added and allowed to stand in the light for 1 hour and no substitution was shown, but when the water was added to the oil solution in carbon tetrachlorid and thoroughly shaken so that the water was taken up by the carbon tetrachlorid, the following results were obtained, the other conditions being the same:

TABLE XVII.—*Bromin absorption with solution of oil in 1 hour.*

Oil solution.	Total.	Addition.	Substitution.	Condition.
Wet..............	217.6	163.9	26.87	Light.
Do...........	166.0	166.0	0.0	Dark.
Dry..............	197.4	197.4	0.0	Light.
Do...........	166.7	166.7	0.0	Dark.

These figures show conclusively that with the bromin solution moisture and light affect most remarkably the reaction between the oil and the bromin. The fact that a little water added to the solution did not affect the results can be explained on the ground that no mixture of the water with the carbon tetrachlorid solution of the oil had taken place. When the water was mixed with the solution and the flask was in the light a substitution of 26.87 was obtained, but when the flask was set in the dark there was no substitution. The addition figures in both cases are practically the same, but do not agree with the addition figures obtained on the dry oil in the light.

This brings us to the conclusion that the bromin solution does not substitute in the light in the absence of moisture, or at least only very slightly; that it does not substitute in the dark even in presence of moisture, and also does not add to the same extent as it does in the light; that the bromin solution in the light and in the presence of moisture and oil forms hydrobromic acid, the amount formed being dependent almost entirely on the time of contact between the oil and the bromin. The blanks of the solutions showed no formation of acid at all. As the solutions of bromin previously used had always given considerable substitution, fresh solutions of bromin, iodin chlorid, and iodin bromid in carbon tetrachlorid were placed in glass-stoppered flasks and used from day to day to see what effect the age of the solution had on the amount of substitution. The results are shown in Table XVIII.

TABLE XVIII.—*Effect of age of solution on iodin numbers and amount of substitution.*

Age of solution.	Bromin.			Iodin chlorid in carbon tetrachlorid.			Iodin bromid in carbon tetrachlorid.		
	Total.	Addition.	Substitution.	Total.	Addition.	Substitution.	Total.	Addition.	Substitution.
	IN LIGHT ONE HOUR.								
Days.									
Fresh.	197.0	197.0	0.0	198.4	194.2	2.20	189.7	189.7	0.0
2	....	....	....	199.2	194.4	2.40	190.0	190.0	0.0
4	195.7	191.2	2.32	197.9	193.2	2.33	190.3	190.3	0.0
6	200.6	189.9	5.41	200.4	198.7	.86	192.3	192.3	0.0
9	196.2	194.5	.86	....	....	....	....	....	....
10	....	....	....	200.6	193.8	3.44	190.0	188.1	.86
13	219.1	162.9	28.16	....	....	....	....	....	....
	IN DARK ONE HOUR.								
13	166.7	166.7	0.0	....	....	....	....	....	....
10	....	....	....	200.4	194.1	3.18	191.1	189.1	.94

Table XVIII shows how much variation there is in the results by the bromin solution when the same solution has been used. The only varying factors are the amount of light due to weather conditions and the amount of moisture which may be absorbed by the carbon tetrachlorid solution of bromin. The absorption of moisture seems to be the only explanation of the great change which takes place on standing. The titer of the bromin solution remains practically constant, showing only a very slight change. That the amount of light may affect the results by the bromin solution to great extent was shown by the following experiments:

The bromin solution in contact with the oil was exposed to direct sunlight for two minutes and a duplicate was kept in the dark two minutes. The total absorption in the first case was 204.7, addition 166.1, substitution 19.30; in the second case the total was 179.7, addition 165.2, substitution 7.29. On the Wijs and Hanus solutions age has very little effect, and it makes no difference whether the reaction takes place in the dark or light as to the amount of substitution or addition when only thirty minutes are allowed for the reaction; if five hours are allowed, as shown in Table XI, light greatly increases substitution.

These results would indicate that in the light with a dry solution bromin will give the same addition numbers as the Wijs and Hanus solutions, but in the dark it does not give addition values which have any relation to those given in the light. The presence of moisture and light gives results of no value at all. If the determinations are made with dry reagents in the dark comparative results can be obtained, but these results can not be compared with figures given by any other methods. From this work it is concluded that the bromin solution is exceedingly unreliable, being affected by conditions hard to control, that the solutions of iodin chlorid and iodin bromid in carbon tetrachlorid are much less affected by these conditions and give much more satisfactory results which are comparable with those obtained by the same reagents in acetic acid, but that these solutions are affected to a greater or less degree by the presence of moisture and for ordinary work would not be satisfactory substitutes for the Hübl method. The following results on moist solutions of oil show that the iodin chlorid and iodin bromid solutions are not affected by the presence of light or moisture in thirty minutes.

TABLE XIX.—*Effect of light and moisture on iodin numbers with iodin chlorid and iodin bromid solutions.*

Solution.	Total.	Addition.	Substitution.	Conditions.
ICl in CCl_4	200.9	192.4	4.25	In light 30 minutes.
Do	200.9	192.4	4.25	In dark 30 minutes.
IBr in CCl_4	191.2	187.7	1.75	In light 30 minutes.
Do	191.2	187.7	1.75	In dark 30 minutes.

The chief use of any solution of this kind is in the detection of such substances as rosin oil and similar products, as the reaction between the oil and halogen is chiefly by substitution, and a solution which gives varied results would be unsatisfactory. The following results on rosin oil show that the Wijs reagent in carbon tetrachlorid is as active as the bromin, and on account of its other advantages would be a much more satisfactory solution to be used for this determination.

TABLE XX.—*Iodin numbers of rosin oil in 30 minutes.*

Solution.	Total.	Addition.	Substitution.
ICl in CCl_4	257.0	5.46	125.7
IBr in CCl_4	190.8	24.50	83.8
Br	257.0	5.46	125.7

The iodin bromid reacts very slowly and gives quite different results from the other solution, while iodin chlorid and bromin give the same results, so that the iodin chlorid would be the most satisfactory solution for this work. But it does not appear that any of these solutions can be satisfactorily substituted for the Wijs, Hanus, or Hübl in the ordinary work, as they give results not comparing well with present data, require many special precautions, and vary in their reaction with age.

Carbon tetrachlorid, which had been recovered by distilling and drying with calcium chlorid and then redistilling, acted much the same as carbon tetrachlorid, which had been shaken up with water, giving large substitution values. We were not able to prepare a recovered product so that it would give the results obtained with Kahlbaum's C. P. dry carbon tetrachlorid.

To illustrate the widely different results that may be obtained by use of carbon tetrachlorid as a solvent in the Wijs and Hanus solutions under the ordinary conditions, several results have been taken from the large number of determinations made in the course of this work. In Table XXI are the results on an olive oil with a Hübl number of 84.6.

Table XXI.—*Iodin numbers on an olive oil with iodin chlorid in carbon tetrachlorid.*

Time.	Total.	Addition.	Substitution.
Minutes.			
5	86.1	85.2	0.45
10	87.3	84.8	2.50
15	87.7	85.2	2.50
30	89.1	85.4	3.70
Hours.			
1	90.8	85.8	5.00
2	94.5	85.0	9.50

The solution used had stood for a few weeks. A fresh solution made up with the same carbon tetrachlorid showed no substitution with linseed oil until it had stood for 1 hour and then very much less than the amount shown in the table with the olive oil. The addition figures, however, remained the same and agreed closely with the Hübl number. The iodin bromid solution in carbon tetrachlorid showed no substitution with olive oils and the amount of substitution with the iodin chlorid solution was very uniform for all the oils. The iodin bromid in carbon tetrachlorid gave higher results than when acetic acid was the solvent, although there was no substitution, while the addition figures with iodin chlorid in carbon tetrachlorid were lower than the figures obtained with acetic acid.

In Table XXII are collected the results obtained on a number of olive oils.

Table XXII.—*Iodin numbers of olive oils by different methods.*

Laboratory. No.	Hübl No.	Wijs No.	Hanus No.	Iodin chlorid in carbon tetrachlorid.			Iodin bromid in carbon tetrachlorid.
				Total.	Addition.	Substitution.	
1	83.7	85.0	83.8	88.1	83.4	2.36	84.8
3	79.9	81.3	79.1	84.8	79.5	2.67	80.8
4	86.1	86.6	85.6	90.3	86.1	2.06	86.9
5	83.2	84.4	83.0	88.0	83.0	2.50	84.3
6	84.6	85.4	84.6	90.2	85.0	2.60	85.7
7	90.6	91.5	89.8	94.5	90.2	2.17	....
10	86.1	87.8	86.7	90.9	85.3	2.82	....

After reviewing all the work done the associate referee offers the following conclusions:

(1) That much better results are obtained by the Wijs and Hanus solutions than by the Hübl.

(2) That the Hanus gives results much more closely agreeing with the existing data and is easier to prepare, but an excess of 60 to 70 per cent is necessary to obtain quick action.

(3) That the Wijs solution is more rapid in its action, and an excess of 35 per cent is sufficient, effecting a large saving in reagents and time of titrating, but it gives higher results.

(4) That 30 minutes is sufficient time for the action of either the Hanus or Wijs solution.

(5) That acetic acid is a better solvent for the work than carbon tetrachlorid.

(6) That the bromin solution or iodin chlorid or iodin bromid in carbon tetrachlorid are not satisfactory for ordinary work.

(7) That iodin chlorid in carbon tetrachlorid is the most satisfactory solution if determination of substitution is to be made.

(8) That both the iodin chlorid and iodin bromid being much less volatile than the bromin, there is much less danger of loss in that way with the former reagents.

RECOMMENDATIONS.

The associate referee offers the following recommendations:

That the Hübl method as given on page 50 of Bulletin 46, Official Methods of Analysis, and on page 24 of Bulletin 65, Provisional Methods of Analysis of Foods, be dropped, and that the following method be substituted, to be called the Hanus method for the iodin absorption of oils and fats.

(A) *Preparation of reagents.*

(1) *Iodin solution.*—(*a*) Dissolve 13.2 grams iodin in 1,000 cc glacial acetic 99.5 per cent acid (showing no reduction with bichromate and H_2SO_4); add enough bromin to double the halogen content determined by titration—3 cc of bromin is about the proper amount. The iodin may be dissolved by the aid of heat, but the solution should be cold when bromin is added.

(2) *Decinormal sodium thiosulphate solution.*—Dissolve 24.8 grams of chemically pure sodium thiosulphate, freshly pulverized as finely as possible and dried between filter or blotting paper and dilute with water to 1 liter at the temperature at which the titrations are to be made.

(3) *Starch paste.*—One gram of starch is boiled in 200 cc of distilled water for 10 minutes and cooled to room temperature.

(4) *Solution of potassium iodid.*—One hundred and fifty grams of potassium iodid are dissolved in water and made up to 1 liter.

(5) *Decinormal potassium bichromate.*—Dissolve 4.9066 grams of chemically pure potassium bichromate in distilled water, and make the volume up to 1 liter at the temperature at which the titrations are to be made. The bichromate solution should be checked against pure iron.

(B) *Determination.*

(1) *Standardizing the sodium thiosulphate solution.*—Place 20 cc of the potassium bichromate solution, to which has been added 10 cc of the solution of potassium iodid, in a glass-stoppered flask. Add to this 5 cc of strong hydrochloric acid. Allow the solution of sodium thiosulphate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste, and with constant shaking continue to add the sodium thiosulphate solution until the blue color just disappears.

(2) *Weighing the sample.*—Weigh about one-half gram of fat or 0.250 gram of oil[a] on a small watch crystal or by other suitable means. The fat is first melted, mixed

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[a] With drying oils which have a very high absorbent power, 0.100 to 0.200 gram should be taken.

thoroughly, poured onto the crystal, and allowed to cool. Introduce the watch crystal into a wide-mouth 16-ounce bottle with ground-glass stopper.

(3) *Absorption of iodin.*—The fat or oil in the bottle is dissolved in 10 cc of chloroform. After complete solution has taken place, 25 cc of the iodin solution are added. Allow to stand, with occasional shaking, for 30 minutes. The excess of iodin should be at least 60 per cent of the amount added.

(4) *Titration of the unabsorbed iodin.*—Add 10 cc of the potassium iodid solution and shake thoroughly, then add 100 cc of distilled water to the contents of the bottle. Titrate the excess of iodin with the sodium thiosulphate solution, which is added gradually, with constant shaking, until the yellow color of the solution has almost disappeared. Add a few drops of starch paste, and continue the titration until the blue color has entirely disappeared. Toward the end of the reaction stopper the bottle and shake violently, so that any iodin remaining in solution in the chloroform may be taken up by the potassium iodid solution.

(5) *Setting the value of iodin solution by thiosulphate solution.*—At the time of adding the iodin solution to the fat, two bottles of the same size as those used for the determination should be employed for conducting the operation described above, but without the presence of any fat. In every other respect the performance of the blank experiments should be just as described. These blank experiments must be made each time the iodin solution is used. Great care must be taken that the temperature of the solution does not change during the time of the operation, as acetic acid has a very high coefficient of expansion, and a slight change of temperature makes an appreciable difference in the strength of the solution. Example blank determinations: (1) Forty cc iodin solution required 62.05 cc of sodium thiosulphate solution. (2) Forty cc iodin solution required 62.15 cc of sodium thiosulphate solution. Mean, 62.1 cc.

Per cent of iodin absorbed:

Weight of fat taken	grams	1.0479
Quantity of iodin solution used	cubic centimeters	40.0
Thiosulphate equivalent to iodin used	do	62.1
Thiosulphate equivalent to remaining iodin	do	30.2
Thiosulphate equivalent to iodin absorbed	do	31.9
Per cent of iodin absorbed ($31.9 \times 0.012 \times 100 \div 1.0479$)		39.61

The following precautions should be exercised in the use of this solution:

1. Great care must be used to prevent change of temperature of the solution, and where any number of determinations are to be made blanks should be measured out at short intervals. This precaution applies as well to the use of the Hübl solution, as the coefficient of the expansion of alcohol is large.

2. When the potassium iodid is added the solution should be thoroughly mixed before the addition of water.

3. The acetic acid must be full strength and pure in order to obtain a solution which will keep well.

The associate referee also recommends that the factor given on page 49 of Bulletin No. 46, Official Methods, for correction for temperature with the refractometer, be changed from 0.000176 to 0.000365 to conform to the correction in Bulletin 65, Provisional Methods, page 22; that under the method on page 32 of Provisional Methods, section 16, in the Halphen test for cotton-seed oil, the brine bath shall boil at from 112° C. to 115° C., and that in making the test for cotton-seed oil the boiling shall be contined for from 1 to 2 hours, the reason for this being that heated cotton-seed oil gives the Halphen test only after long heating, heating from 15 to 30 minutes giving practically no reaction.

For Renard's test[a] for peanut oil, as modified by Tolman, page 33, paragraph 18, Bulletin No. 65, Provisional Methods, the following modified method is recommended:

Weigh 20 grams of oil into an Erlenmeyer flask. Saponify with alcoholic potash, neutralize exactly with dilute acetic acid, using phenolphthalein as indicator, and wash into a 500-cc flask containing a boiling mixture of 100 cc of water and 120 cc of a 20 per cent lead acetate solution. Boil for a minute, and then cool the precipitated soap by immersing the flask in water, occasionally giving it a whirling motion to cause the soap to stick to the sides of the flask. After the flask has cooled, the water and excess of lead can be poured off and the soap washed with cold water and with 90 per cent (by volume) alcohol. Now add 200 cc of ether, cork the flask, and allow to stand for some time until the soap is disintegrated, then heat on the water bath, using a reflux condenser, and boil for about 5 minutes.[a] In the oils most of the soap will be dissolved, while in lards, which contain is so much stearin, part will be left undissolved. Cool the ether solution of soap down to from 15° to 17° C., and let stand until all the insoluble soaps have crystallized out—about twelve hours are required.

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[a] Renard, Comp. rend., 1871, **73**: 1330. Benedikt and Lewkowitsch, Oils, Fats, and Waxes, p. 365.

Filter and thoroughly wash the precipitate with ether. Save the filtrate for the determination of the iodin number of the liquid fatty acids by the Muter method. The soaps on the filter are washed back into the flask by means of a stream of hot water acidified with hydrochloric acid. Add an excess of dilute hydrochloric acid, partially fill the flask with hot water, and heat until fatty acids form a clear, oily layer. Fill the flask with hot water, allow the fatty acids to harden and separate from the precipitated lead chlorid; wash, drain, repeat washing with hot water, and dissolve the fatty acids in 100 cc of boiling 90 per cent (by volume) alcohol. Cool down to 15° C., shaking thoroughly to aid crystallization. From 5 to 10 per cent of peanut oil can be detected by this method, as it effects a complete separation of the soluble acids from the insoluble, which interfere with the crystallization of the arachidic acid. Filter, wash the precipitate twice with 10 cc of 90 per cent (by volume) alcohol, and then with alcohol of 70 per cent (by volume). Dissolve off the filter with boiling absolute alcohol, evaporate to dryness in a weighed dish, dry and weigh. Add to this weight 0.0025 gram for each 10 cc of 90 per cent alcohol used in the crystallization and washing if done at 15° C.; if done at 20°, 0.0045 gram for each 10 cc. The melting point of arachidic acid obtained in this way is between 71° and 72° C. Twenty times the weight of arachidic acid will give the approximate amount of peanut oil present. No examination for adulterants in olive oil is complete without making the test for peanut oil.

The associate referee wishes to acknowledge the assistance received from A. F. Seeker, of the laboratory of the Bureau of Chemistry, who practically repeated all of the work in checking up the results obtained, although in the bromin work it was found almost impossible to duplicate any set of results.

In continuation of the report on fats and oils the following paper was submitted by the associate referee:

REPORT OF COOPERATIVE WORK ON THE DALICAN TITER TEST.

By L. M. Tolman, *Associate Referee on fats and oils.*

Work on this test was begun at a very late date, and this report can be considered only as as a preliminary one presented with the idea that by the next meeting of the association some definite and satisfactory method may be offered.

The various modifications of the test in use do not give a satisfactory agreement, and several of the largest concerns in this country using it expressed a desire that an effort should be made by the Association of Official Agricultural Chemists to cooperate with them, as we have with the tannin industry, and prepare uniform methods of analysis so that results obtained by different analysts shall agree. In tests of this kind, which are to a certain degree empirical, uniformity of methods, even as to details, is absolutely essential.

The first step was to send samples of fats to the various men interested, requesting them to report their results and to give in detail their methods. Results were received from 19 chemists representing the following firms in which the test is in constant use: American Cotton Oil Company, Southern Cotton Oil Company, Armour & Co., Nelson Morris & Co., James Kirk & Co., Colgate & Co., Procter & Gamble, Stillwell

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[a] Process used by N. J. Lane in his modification of Muter's method. J. Am. Chem. Soc., 1893, 15: 110.

& Gladding, W. J. Wilcox Lard and Refining Company, Central Lard Company, and Swift & Co. Results were also received from Prof. A. H. Gill, of the Massachusetts Institute of Technology, and G. E. Dyck, of the National Provisioner Laboratory.

The following directions for reporting results were sent with the samples of fat:

State the detail of the method used, also size and thickness of test tube used, and capacity of bottle.

Give the saponification value of the fatty acids.

Give the temperature of the room at time of test.

If possible have thermometer used checked against a standard, also state kind and dimensions of thermometer used, size of bulb especially.

If possible be present at the meeting of the Association of Official Agricultural Chemists, November 19, 1903, in order that there may be a thorough discussion of the results, and a satisfactory method outlined for next year.

Very complete reports were sent in by practically all of the analysts and a large amount of valuable data was thus obtained. It was found from these reports that there are a number of methods of procedure in use in this country. These can be brought under three heads: (1) Modifications of Dalican's method; (2) modifications of Wolfbauer's method; (3) modifications of Finkener's method. As it has long been known that these different methods give varying results it was not expected that the figures obtained would agree closely.

The following modification of Dalican's test, used by James Boyce, of the American Cotton Oil Company, and David Wesson, of the Southern Cotton Oil Company, was practically the same as that employed by 10 of the chemists:

APPARATUS.

A round iron or metallic dish, 1.5 liters capacity.

A porcelain dish, 2 liters capacity.

Test tube, 22 mm to 25 mm diameter, 15 cm long, the walls of the tube to be 1.5 mm thick.

One bottle, 7.5 cm. diameter, 15 cm. deep, to have wide mouth, with cork of sufficient size to hold test tube. Bottle to be of clear glass, with thick wall.

A delicate certified thermometer, reading in $\frac{1}{10}$ degrees, graduated from 0° to 70° C. Bulb of thermometer to be not more than 6 mm. in diameter and 3.4 cm. long.

An accurate thermometer to hang alongside of the apparatus to record the temperature of the room in which test is made.

OPERATION.

Weigh 50 grams of the fat into a metal dish. Measure 40 cc of caustic soda, 36° Beaumé, and 50 cc of 95 per cent alcohol into the small bottle. Heat the fat over a gentle flame to 115° to 120° C. Shake the alcohol and soda solution well together and add to the fat. Stir vigorously over a gentle fire until soap becomes sufficiently dry to form pieces which will no longer adhere to the spatula by gentle pressure.

Redissolve in 20 cc of alcohol and again dry to a condition in which the soap will not adhere to the spatula by pressure.

Transfer the dry soap to the porcelain dish. Add one or two liters of boiling water and keep the mixture boiling for 40 minutes. Replace, by adding cold water, about the same quantity of water which has been evaporated by the previous boiling. Add to the soap solution 70 cc of 25° Bé. sulphuric acid. Boil again until the fatty acids, which will float on top, are perfectly clear and transparent and show no signs of any particle of soap which has not dissolved. Siphon off the acid water and transfer the fatty acids to a small beaker, melt the acids and allow the water to settle; transfer to a dry beaker, dry in a water bath or on a steam bath for 20 minutes and filter the fatty acids through white filter paper, using a hot-water funnel.

Place the clear fatty acids in the titer tube, filling it two-thirds full, the tube being first warmed in the water bath. Insert the tube in the perforated cork of the large bottle, to serve as an air bath, and hang the delicate thermometer so that the bulb will be exactly in the middle, in all directions, of the mass, and allow the fatty acids to cool until the mercury stops falling and just begins to rise again. Then move the thermometer rapidly around the sides of the tube, three times to the right and three times to the left, without any up or down motion, finally replacing the thermometer in the center of the tube.

Watch the thermometer as the mercury rises and take the highest point reached as the titer test, at the same time recording the temperature of the room in which the test was made.

The results on the three samples of fat obtained by this method are collected in Table I.

TABLE I.—*Results obtained by modifications of Dalican test.*

Analyst.	Titer.			Test tube.		Bulb of thermometer.		Method of stirring.
	Sample No. 1.	Sample No. 2.	Sample No. 3.	Diam.	Length.	Diam.	Length.	
Sent by James Boyce, American Cotton Oil Co., Chicago:	° *C.*	° *C.*	° *C.*	*cm.*	*cm*	*cm.*	*cm.*	
J. B	49.6	40.15	36.30	2.2	15	0.6	3.5	3 times to right and 3 times to left.
C. C. B	49.9	40.3	36.2	2.2	15	.6	3.5	
C. B. C	49.35	40.4	36.33	2.2	15	.6	3.5	
J. H. S	49.40	40.15	36.43	2.2	15	.6	3.5	
A. D. W	49.5	40.40	36.2	2.2	15	.6	3.5	
Sent by David Wesson, Southern Cotton Oil Co.:								
R. E. H	49.6	40.8	36.2	2.5	15	......	......	Do.
D. W	49.6	40.4	35.9	2.5	15	......	......	
W. J. B	49.7	40.5	36.3	2.5	15	......	......	
L. M. Tolman, U. S. D. A	49.3	40.3	36.0	2.2	15	.5	2.0	Do.
A. F. Seeker, U. S. D. A	49.55	40.3	35.75	2.2	15	.5	2.0	Do.
E. G. Halloway, James Kirk & Co.	49.50	40.0	35.90	1.7	15	.4	3.8	Do.
R. D. Oilar, Wilcox Lard Co	49.58	40.35	36.30	2.5	15	.6	2.3	Do.
E. K. Nelson, Nelson Morris & Co.	49.85	39.95	35.9	3.0	18	.6	4.5	Do.
Theo. Smith, Central Lard Co.	49.45	40.20	35.65	2.5	21	......	3.0	Do.
A. H. Gill, Massachusetts Institute Technology.	49.50	39.8	37.6	1.2	15	.35	1.7	
Stillwell & Gladding	49.9	40.7	36.7	......	......	......	......	6 times to right and 6 times to left.
W. D. Richardson, Swift & Co.	49.7	40.7	36.6	......	......	.3	1.5	
Maximum	49.9	40.8	37.6	......	......	......	......	
Minimum	49.3	39.8	35.65	......	......	......	......	
Average	49.5	40.2	36.1	......	......	......	......	
Difference between maximum and minimum.	.6	1.0	1.95	......	......	......	......	

The results in Table I show a fair degree of uniformity, although the details of carrying out the method varied somewhat. But if the results of those using test tubes 2.2 to 2.5 cm in diameter and stirring the acids three times to the right and three times to the left are compared, a much more satisfactory agreement is shown. Twelve sets of results come under this head and the comparison is as follows:

TABLE II.—*Results obtained with uniform test tubes and stirring.*

Determinations.	Sample No. 1.	Sample No. 2.	Sample No. 3.
	° *C.*	° *C.*	° *C.*
Maximum	49.9	40.8	36.4
Minimum	49.3	40.2	35.6
Difference	.6	.6	.9

There are a number of other factors which influence titer, such as moisture and kind of thermometer, and while the above results are fairly satisfactory much better ones would undoubtedly be obtained if uniform methods of drying the fatty acids were used and uniform thermometers. Some of the thermometers used were very long and the correction for the mercury out of the fat would be considerable. Wolfbauer found that the method of saponification did not influence the titer, providing the alcohol was completely removed in alcoholic saponification, but that moisture had a marked influence and titers 0.6 lower were found in moist than in dry fats. He also found that the size of the test tube affected the results, a 2½-cm tube giving results 0.2 lower than a 3½-cm tube, although tubes wider than 3½ cm did not affect the results. These are points which should be considered in our future work.

The details of the work of the various chemists who used the Dalican method will be given in so far as they differ from the method given above. Mr. Boyce and his associates, Mr. Wesson and his associates, Mr. Oilar, Mr. Seeker, and the associate referee followed this method in detail. E. G. Halloway, of James Kirk & Co., makes a slight change. He begins to stir three times to the right and three times to the left when incipient solidification sets in.

E. K. Nelson, of Nelson Morris & Co., gives the following directions:

> Weigh out 50 grams of fat into an 8-inch porcelain dish. Melt on a steam bath and saponify with a mixture of 30 cc of 40° Bé. caustic soda and 50 cc. methyl alcohol. * * * Boil until the fatty acids are perfectly clear, collect them, and dry them for two hours at 100° C. * * * Place the thermometer bulb as nearly as possible in the center of the fatty acids and watch the mercury column until it becomes stationary. * * * Then raise the cork from the test tube and stir the fatty acids by moving the thermometer three times in one direction and three in the opposite direction around the wall of the test tube.

Mr. Nelson also makes the following suggestion from his experience:

> While with Swift & Co. in the capacity of chief chemist, I observed a discrepancy between two laboratories which was traced to the fact that one chemist used a quick-acting thermometer, short stem with small bulb, while the other chemist used a long thermometer with a large bulb.

Theo. Smith, of the Central Lard Company, makes the following comment:

> Any moisture clinging to the cake can be removed with a piece of filter paper, but on no account should the acids be dried between filter papers, as is done by some analysts who obtain results a degree or so above anyone else. Nor should the acids be dried in an air bath, as mixtures containing the acids of cotton-seed oil and other unsaturated acids susceptible to oxidation often give abnormal results if so treated. * * * A few drops of water in the acids make no difference with the titer as they settle to the bottom of the tube and do not interfere with the crystallization.

Prof. A. H. Gill, of the Massachusetts Institute of Technology, followed Dalican's method as given in Lewkowitsch.[a]

Stillwell & Gladding use the French Syndicate method, which is a modification of Dalican's test, and give the following additional modifications:

> Decant the clear, fatty acids into a shallow flat-bottom dish, about 4 inches in diameter, and heat for 20 minutes in a water oven at 100° C. This expels the last traces of water, and is not prolonged enough to affect a true titer test.
>
> In stirring with the thermometer at the point where the mercury remains stationary, stir vigorously half a dozen times from right to left, and the same number from left to right. In doing this be careful neither to raise nor lower the thermometer. The object of this is to avoid mixing with the fat acids in the neighborhood of the bulb of the thermometer, the fat acids above the bulb which are hotter, or the fat acids below the bulb which are colder.

The result of our work would lead one to favor the method of constant stirring as the best method for making titer determinations were it not for the fact that in the oleo-stearine sample the constant stirring method seems to give too low results. This is attributed to the fact that the stearine (crystallizing at so high a temperature) cools off so rapidly that the constant stirring keeps the whole mass below the proper point. When this oleo stearine was tested by the constant-stirring method at a room temperature of 100° F. a reading of 49.90° C. was obtained.

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[a] Second ed., p. 133.

If, however, the stirring is maintained until the mercury stops and just begins to rise, and the thermometer is then allowed to stand quietly in the center of the tube the proper titer point is obtained.

TABLE III.—*Results obtained by Stillwell and Gladding.*[a]

Determination and method.	Oleo stearine.	Edible tallow.	White grease.
Saponification value	204	204	198
	° *C.*	° *C.*	° *C.*
Titer (French Syndicate method)	49.90	40.70	36.70
Titer (constant stirring)[b]	49.70	40.70	37.10
Titer (constant stirring till mercury stops falling and just begins to rise, then stop stirring, and watch mercury as it rises)	49.90	40.90	37.10

[a] Temperature of the room about 70° F.

[b] Using same tube as in French Syndicate method and stirring with horizontal circular motion, without any up and down motion.

The third method appears to be the best one for determining the titer.

Wilson H. Low, of the Cudahy Packing Company, waits until the crystals in the tube begin to surround the bulb of the thermometer, then stirs. He makes the following comment:

> We buy fat of other parties who make the test in a larger tube and with continual stirring, and their method makes the titer about a degree higher than ours. As the test is only an empirical one it doesn't make much difference how it is made, but it is desirable for trade purposes that there should be some standard, so that it can be known definitely what is meant when a fat of a stated titer is offered.

Mr. David Wesson reported as follows regarding several different methods of saponification and stirring:

> Experiments were planned to determine, first, the effect of two different methods of saponification; second, the effect of reading in different size tubes and different size air baths; third, results obtained by different methods of stirring. These three points seem to be the cause of a great many discrepancies between different observers. Owing to continued warm weather most all the observations were made at room temperatures running from 23° to 28° C. The following methods were employed for saponification:

Method No. 1 for saponification.

> Fifty grams fat, 40 cc caustic soda, specific gravity 1.318, 50 cc alcohol 95 per cent, heated together in a 10-inch porcelain evaporating dish on a steam bath. The fat did not reach a temperature above 94° C. The alcohol was evaporated off and the soap run to dryness as shown by breaking up the lumps which no longer stuck to the glass rod.

Method No. 2—Dalican's method.

> *First method of taking setting point.*—Titer as in Dalican's method and use a large tube 25 mm in diameter and an air bath of 1,200 cc capacity.

TABLE IV.—*Results obtained by three analysts with method No. 1.*

No. of sample.	R. E. N.		D. W.		W. H. B.	
	Titer.	Room temperature.	Titer.	Room temperature.	Titer.	Room temperature.
	° *C.*	° *C.*	° *C.*	° *C.*	° *C.*	° *C.*
1	49.9	29	49.9	29	49.8	26
[a]1	49.6	26	49.6	27	49.7	26
2	40.3	24	40.4	28	40.2	26
[a]2	40.8	27	40.4	26	40.5	26
3	35.8	23	36.2	25	36.3	26
[a]3	36.2	28	35.9	25	36.3	26

[a] Saponified by Dalican's method.

Second method of taking setting point.—Same tubes and bath were used as in method No. 1. The thermometer was stirred slowly, about 60 turns per minute, and continuously until the mercury ceased dropping. The bulb of the thermometer was then left in the center of the mass, and the highest point reached by the mercury was noted.

TABLE V.—*Results obtained by two analysts with method No. 2.*

No. of sample.	D. W.		W. H. B.	
	Titer.	Room temperature.	Titer.	Room temperature.
	° *C.*	° *C.*	° *C.*	° *C.*
1	50.2	27	50.3	29
[a]1	50.2	27	50.2	28
2	41.0	27	41.2	29
[a]2	41.2	27.5	41.4	29
3	37.3	25	37.5	29
[a]3	37.2	25	36.1	28

[a] Saponified by Dalican's method.

Third method of taking setting point.—The readings were taken in the same manner as in methods Nos. 1 and 2, except that the air bath had a capacity of 500 cc, and the titer was taken in ordinary test tubes 18 mm in diameter. The results given below show the effect of cooling without stirring, except at the end of the operation, and of cooling by constant stirring.

TABLE VI.—*Results obtained by method No. 3 (W. F. B.).*

No. of sample.	Titer. (Cooled without stirring, except at end of operation.)	Room temperature.	Titer. (Cooled by constant stirring.)	Room temperature.
	° *C.*	° *C.*	° *C.*	° *C.*
1	49.7	26.6	50.2	26.8
[a]1	49.7	27.5	50.0	27.4
2	40.2	27.0	41.0	27.5
[a]2	40.4	27.0	41.2	26.8
3	36.2	27.6	37.3	27.5
[a]3	36.2	27.0	36.3	27.0

[a] Saponified by Dalican's method.

Examination of the results seems to show that at the temperature at which the work was done the size of test tube used had no effect.

(2) Higher results were obtained in most cases by constant stirring.

(3) It was noted in making the experiments that it was harder to get definite readings on the soft fat sample No. 3 by constant stirring than by allowing the fat to cool down slowly as in method No. 1.

Three chemists used modifications of Wolfbauer's method.

TABLE VII.—*Results obtained by modifications of the Wolfbauer test.*

Analyst.	Titer of samples Nos.—			Size of test tube.		Size of thermometer bulb.	
	1.	2.	3.	Diameter.	Length.	Diameter.	Length.
	° *C.*	° *C.*	° *C.*	*cm.*	*cm.*	*cm.*	*cm.*
M. H. Ittner, Colgate & Co	50.47	41.43	37.6	2.6	7.5	0.5	3.0
J. E. Weber, Procter & Gamble	50.00	40.60	....	4.5	11.0	.4	2.3
David Wesson	50.20	41.20	37.2	2.5	15.0	....	....

It will be seen that this method gives higher results than the Dalican method. The details of the operation in Armour & Co.'s laboratory are given by A. G. Manns, as follows:

Seventy-five grams of the fat are placed in an iron dish with 150 cc of caustic soda lye of 13 per cent to 14 per cent strength. The mixture is boiled to dryness with constant stirring so as to avoid scorching. In practice, a 3-pound lard pail is used instead of the iron dish and a heavy stirring rod to break up the lumps. The dry granular soap is then transferred to a porcelain dish of about 1,000 cc capacity, in which 500 cc of water and 250 cc of sulphuric acid of 60 per cent strength have been previously mixed. The whole is boiled until the soap is decomposed, water being added from time to time to maintain the original volume. An enameled cover is kept on the dish while boiling. When the fatty acids are perfectly transparent they are washed several times with boiling hot water, allowed to settle, decanted, filtered through several thicknesses of dry filter paper, and dried for four hours at 105° C. The fatty acids when dried are cooled to about 15° or 20° C. above the expected titer and transferred to a special tube 1 inch in diameter and 4 inches in length. The tube is made of glass about $\frac{3}{64}$ of an inch thick. It is placed in a bottle fitted with a cork which is perforated so as to hold the tube rigidly when in position. The thermometer, graduated to 0. 1° C., is suspended so that it can be used as a stirrer, and the mass is stirred slowly until the mercury remains stationary for at least 30 seconds. The thermometer is then allowed to hang quietly with the bulb in the center of the mass and the rise of the mercury is observed. The highest point to which it rises is taken as the titer of the fatty acids.

The fatty acids should always be tested for complete saponification as follows:

About 3 cc of the fatty acids are placed in a test tube and about 15 cc of strong alcohol added, the mixture brought to a boil, and an equal volume of ammonia, specific gravity 0.96, added.. A clear solution should result, turbidity indicating presence of unsaponified fat.[a]

It will be noted that the method differs from the Dalican test in two important details. First, the fatty acids are thoroughly dried; second, the method gives a definite time when to stop stirring. The thorough drying of the fatty acids was adopted as given above, as it has been clearly demonstrated that moisture in the fatty acids lowers the titer about 0.4° to 0.5° C. Further, if the fatty acids are dried in hydrogen gas by heating up to 150° C., the titer is the same as if the heating was performed in the open air, or if they were dried in a bath for about four hours at 105° C.[b]

Mr. Ittner uses the following method:

The acids are detached and the dish drained of nearly all its water and the acids melted down in the same dish. After the acids are melted clear, they are poured into a dry bottle and kept on the water bath until the titer can be taken.

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[a] Lewkowitsch, Chem. Anal. of Oils, Fats, and Waxes, 2d ed., p. 100.

[b] Wolfbauer's work, Lewkowitsch, Chem. Anal. of Oils, Fats, and Waxes, 2d ed., p. 134.

The acids in the test tube are cooled down by holding the tube in the hand and stirring with the thermometer. When the temperature falls within a degree or two of the point of incipient crystallization and while they are still clear and fluid, the tube is put into the outer jar and the stirring carefully continued without intermission until very fine crystals begin to form throughout the mass. Then the stirring is stopped and the bulb carefully adjusted to the center of the acids. At this point, there should be no thick masses of crystals, but only a few fine scattered ones. Crystallization started in this way proceeds evenly throughout the mass. The temperature may fall a little, then it usually begins to rise again until a maximum is reached, at which point the reading is constant for several minutes. The mercury then begins to fall, and the maximum temperature reached just previous to this is taken as the titer test. The acids are evenly solidified around the outer parts of the test tube, but the inner portion around the bulb is still quite soft.

One chemist, Mr. G. E. Dyck, of the National Provisioner Laboratory, used a modification of Finkener's method and his results are given below:

TABLE VIII.—*Titer on official samples by Finkener's method.*

Titer of samples Nos.—			Size of test tube.	
1.	2.	3.	Diameter.	Length.
°*C.*	°*C.*	°*C.*	*cm*	*cm.*
49.85	40.7	37.35	3.8	10.0

These results are slightly higher than those given by the Dalican method and not so high as those by Wolfbauer's method.

The special features of Mr. Dyck's method are as follows:

When the acids are clear, the underlying water is withdrawn by means of a siphon or the filter pump, fresh, cold water is added and the acids liquefied again until clear. The water is again removed and the solid acids washed twice with cold water and dried with unprinted newspaper, whence they are placed in a beaker and the latter in the water bath. When perfectly liquefied the acids are poured off the remaining water into a dry beaker and once more heated in the bath for 30 minutes. The acids are next filtered through a dry, folded filter paper into a tube 1½ by 4 inches in the drying oven at a temperature of approximately 15° to 20° above the supposed titer. The tube is placed in a paper mailing case and this in a wooden box 3 by 3 by 6 inches, provided with a close-fitting lid, according to Finkener, the empty space being filled with absorbent cotton. The whole is previously placed in the drying oven and warmed to the temperature of the filtering acids.

The perforated lid of the box is closed and at the appearance of the first crystals at the bottom of the tube the acids are stirred by the thermometer to the right and left a few times. When the mercury stops falling the stirring is repeated so that the crystals are uniformly distributed throughout the entire mass, without, however, touching the sides of the tube with the thermometer. The stopping point is noted as well as the high point reached by the mercury, but the latter only is reported.

It is evident from this work that there is need of a uniform method of procedure if agreeing results are to be obtained. The reports show that the two methods in general use are Dalican's and Wolfbauer's, and it was thought by those present at the meeting that these two methods would furnish a satisfactory working basis for the coming year, the idea being that these methods should be tried and the one giving the most satisfactory results be recommended for adoption as the official method.

The PRESIDENT. If there is no discussion, we will take up the subject of fruits and fruit products.

REPORT ON FRUITS AND FRUIT PRODUCTS.

By L. S. Munson, *Associate Referee.*

The directions given in Bulletin No. 65, Provisional Methods of Food Analysis, page 78, for the polarization and determination of cane sugar, differ from the method for these operations to be submitted at this meeting, by the referee on sugar, as the official method. I recommend, therefore, that the methods on page 78, under fruits and fruit products, for polarization and the determination of cane sugar be made to conform to the official sugar methods, that there may not be two different methods for making the same determination. For the determination of reducing sugars the Allihn method is given (sec. 14, p. 78), for which should be substituted the regular official method, which is the Meissl method used with the Meissl and Hiller tables, and this method should be used also for the determination of cane sugar by the process of inversion.

On page 78, section 15, is given a method for the determination of dextrin. The only object in determining dextrin in fruit products is to arrive approximately at the amount of glucose in the materials. Since Bulletin 65 was published, two years ago, a method has been suggested by A. E. Leach, and another by Edward Gudeman, for the determination of commercial glucose in mixtures of this nature. Mr. Leach's method is printed as provisional on page 48 of Bulletin No. 65. Mr. Gudeman's method may be found on page 65 of the Nineteenth Annual Proceedings of this association for 1903, Bulletin No. 73. I believe that both of these gentlemen have some remarks to make concerning these methods. I have no further recommendations to make concerning fruits and fruit products.

The **President**. I suggest that we take up the subject of saccharine products in order to give the gentlemen an opportunity to present the papers or remarks on glucose.

THE DETERMINATION OF COMMERCIAL GLUCOSE IN SOME SACCHARINE PRODUCTS.

By Albert E. Leach.

The determination of commercial glucose in such products as molasses and maple sirup, wherein the amount of invert sugar is comparatively small, is readily accomplished by some such means as the provisional method already given in Bulletin 65, but its determination in such products as honey, jams, and jellies presents some obstacles.

At first sight it would seem to be only necessary to divide the invert polarization of the sample at 87° C. by the assumed factor for the polarization of commercial glucose to obtain the percentage of the latter. As a matter of fact, a normal solution of commercial glucose polarizes somewhat lower after than before inversion, and the reading at 87° is very much lower than that at 20°. The difference between the readings at the high and low temperature may be due in part to expansion, but another disturbing influence enters in to affect the results, namely, the action of the acid used in inversion on the maltose and the dextrin of the glucose at high temperatures.

If a normal solution of commercial glucose be subjected to inversion in the ordinary manner, it will be found impossible to obtain a constant reading at a temperature of 87° C. The polarizing figure drops quite rapidly, due no doubt to the hydrolyzing action of the hydrochloric acid on the maltose and the dextrin. On this account it becomes necessary, when analyzing samples of honey, jams, and jellies containing commercial glucose, to neutralize the sample as soon as possible after inversion, before the hydrolytic action of the acid has set in to any appreciable extent.

It is found best to prepare separate normal solutions of the sample on one of which the direct polarization is obtained in the usual manner. For the invert readings the second solution is prepared as follows: Weigh out half the normal weight of the sample, 13.024 grams, in a 100 cc graduated flask. Dissolve in about 70 cc of water and add 7 cc of concentrated hydrochloric acid. Then heat to 68° C. and cool in the usual manner for inversion. Add a few drops of phenolphthalein, and enough sodium hydroxid to neutralize. Discharge the pink color with a few drops of dilute hydrochloric acid, cool again, add from 5 to 10 cc of the appropriate clarifier, which in the case of honey would be alumina cream, and make up to the mark. Constant and presumably correct readings may be made with this solution both at room temperature and at 87° C. using the 200 mm tube and multiplying the readings by two.

Following are the results of a series of readings made on five samples of commercial glucose by H. C. Lythgoe, showing the relation between the two invert readings at 22° and 87°, respectively, as well as the ratio of the invert polarization at 87° to the direct polarization. The readings were made on solutions subjected to the above method of inversion and neutralization.

TABLE I.—*Polarizations of 5 samples of commercial glucose.*

Source.	Density.	Polarization.				
		A Direct.	B Invert at 22° C.	C Invert at 87° C.	Ratio of C to B.	Ratio of C to A.
Boston Molasses Co.:	°*Bé.*					
No. 1	42	156.6	153.4	146.6	0.956	0.936
No. 2	42	158.6	154.6	149.0	.964	.940
Illinois Sugar Refining Co	42	169.6	165.4	159.4	.964	.940
New England Confectionery Co	43	167.4	162.8	155.0	.952	.926
Illinois Sugar Refining Co	45	174.0	171.0	161.2	.943	.927
Average					.956	.933

Obviously, then, if we are to express results in terms of commercial glucose, polarizing at an assumed figure (for example, 175 as in the provisional method), it will be necessary in the case of jellies and jams to divide the high reading at 87° C. after inversion by 93 per cent of the assumed factor, or if 175 is the factor by 163. It is obvious also that the calculation of invert sugar in products which contain also considerable commercial glucose is a difficult one, by reason of the influence of the commercial glucose on the polarization. We are at present working on this problem and hope before long to be able to work out a reliable formula for the calculation of invert sugar in the presence of commercial glucose.

It has been thought best, however, at this time to call attention to these peculiarities of commercial glucose, in view of the fact that it is understood that the provisional method, while well adapted for molasses and sirups, is being quite largely used for jams and jellies as well. No doubt a rough approximation can be made by the use of the unmodified provisional method for all these products, but where verification of manufacturers' formulas is involved, or where more exact methods are needed, the above precautions should be borne in mind.

The PRESIDENT. We are indeed fortunate in having with us this morning one who is doing so much for the agricultural interests of the country and who has always taken a deep interest in the work of our association. It affords me great pleasure to introduce the honorable Secretary of Agriculture, Mr. James Wilson, to the association. (Applause.)

ADDRESS OF SECRETARY WILSON.

Mr. President and Gentlemen of the Association: The Secretary of Agriculture can do very little; one man can do very little anywhere, unless he is well sustained in his efforts along the line of his work. I want to say to you that it has been a matter of great satisfaction to myself and the Department of Agriculture to have such steady, unflagging support as we have had from this association. We regard you as among our most intelligent friends. The work that you have been doing has been of great value to the country and is beginning to bear fruit.

We are having a good measure of success in executing the law with regard to the importation of foreign foods that are not good enough for the people who live in the countries where they originate. The conclusion has been reached that they are not good enough for us, and new light is being brought to the subject daily in that the merchants who import those goods are learning that they need the protection of the American chemists. They do not know when goods are adulterated; at least, they have not known, but they are discovering it now. The law is being successfully put in force and the merchants of the United States give it a hearty welcome, and it will work a complete cure in a few years.

I am well satisfied that the American merchants will not endeavor to surreptitiously bring into the United States things that should not be brought in—that are contrary to law. I believe that a very large proportion of the American people are honest people. But we have to admit that once in a while we have a rascal among us and he has to be looked after a little—but I believe he is the extreme exception.

We must take another step. The States are not able to protect themselves and enforce their food laws beyond their own borders. I really think we have come to the time when the Federal Government should give us interstate pure food laws, so that our people may be protected from an occasional rascal we may have at home. I hardly think that we can claim that nobody in the United States is disposed to cheat his neighbor; we are not that near the millennium yet. But this association is aiding us to reach conclusions which I think will help us to discipline the people at home who require discipline. I believe the time has come, and the steady pressure exerted by the gentlemen of this association has brought it about to a large extent, when we should look after the movement of these matters in their relation to interstate commerce.

I have only a moment and you are busy people. I only came to take my hat off to you and give you a hearty welcome and say good day. (Applause.)

The discussion on glucose methods was then resumed.

Mr. Leach. Mr. Gudeman and I have had a friendly controversy with reference to glucose methods. His method is based on the hydrolysis of sugars and mine on the simple polarization. When dealing with such a complex substance as commercial glucose it is impossible to get an exact method for its determination in saccharine products, as its moisture content, polarization, and density all vary. But at the same time we are confronted with the fact that it is necessary sometimes in court work and in verifying formulas on packages for the chemist to adopt some method and express the results in percentage, no matter how unsatisfactory they may be. Therefore, I think the method chosen should be accepted subject to its limitations and the results given in the terms of some arbitrary factor. The factor assumed in my formula, i. e., the highest polarization of a given grade of commercial glucose used by dealers that, within our experience, has ever been found, gives the manufacturer the benefit of the doubt in every case. If we find 75 per cent of glucose the chances are that the manufacturer has used about 80 per cent. That is, he has used a lower polarizing glucose than we credit to him. At best this is unsatisfactory, but from the commercial standpoint the ease with which the amount of glucose is calculated should be considered. The sample must be polarized in any case, and having done this, the per cent of glucose may be calculated by the formula and the facts obtained quite as easily as by Mr. Gudeman's method based on the hydrolysis of sugars.

Mr. **Gudeman.** The method which I suggested was not offered as a provisional method, but simply as a method giving absolute results. If you compare it with Mr. Leach's method you will see that his method has all the advantages of rapidity, simplicity, and cheapness. But when it comes to absolute accuracy, the method I suggested of determining the component parts of glucose gives closer results. At the same time a very slight modification of Mr. Leach's method will lead to these results, and that modification consists in accepting a constant for the polarization of glucose, and also defining the percentage or accepting a constant for the percentage of water in glucose, and using these two factors irrespective of the article under examination. The whole gist of my original paper is found in a few lines, on page 68, of the proceedings of this association for last year, where I stated that the use of different factors of rotation, subject to the samples under examination, will lead to greater errors than the use of a constant (175), even if the same is not correct. The main criticism I have to make of Mr. Leach's method is that when he examines certain products the constant 175 is used, for other products 150, and in another case 185. Now, if this association will simply define glucose in terms of percentage of water, irrespective of whether it is on a basis of dry substance or the maximum limit of water, 22.5 per cent, and set a standard of polarization, the results obtained by different chemists will agree. As it is now they do not agree. Some chemists are using Mr. Leach's method, and others, among them official chemists, are using the factor 175 for all products that contain glucose. If you recalculate the percentages published you will see that the factor is always between 175 and 176. Mr. Leach is under the impression that various grades of glucose are manufactured for various purposes. That is not so. There is only one kind of glucose manufactured in the United States to-day, but there are different degrees of moisture and the polarization is affected thereby.

I would suggest that this association adopt a constant. I did not come prepared with a paper, but wanted to bring out the fact, and it has been brought out, that the methods of determining glucose are now under consideration by the various chemists, and I believe it will not be long before a method is adopted that will give at least comparative results by different analysts.

Mr. **Wiley**. What Mr. Gudeman has said in regard to this matter is without question, assuming that his statement, that there is only one grade of glucose made in the United States which varies only in the percentage of water present, is a correct one. I do not mean to accuse Mr. Gudeman of making incorrect statements, but that would be a very difficult thing to secure. It is true that glucose is practically made under the direction of a single firm, the Corn Products Company, which undoubtedly always uses the same processes, and hence his statement comes nearer being true now than ever before in the history of glucose making.

When, in 1880, glucose first became a prominent element in commerce, I was called upon to make some examinations of this substance, and discovered that there was a constant relation between the reducing power of glucose on copper salts and its polarizing power. If all glucoses are reduced to the same standard of water or calculated to the dry substance, you will find an exact relation between the copper-reducing power and the polarization. So exact is this relation that you can polarize a glucose and calculate the amount of copper it will reduce, or vice versa. But even under a single direction there must be differences in the amount of conversion in the starch. It is not technically possible to stop every conversion at exactly the same point. That is the only flaw in Mr. Gudeman's reasoning. If the conversion or hydrolysis were always carried to the same point, then a constant factor could be adopted which would be perfectly satisfactory. But otherwise, a constant factor can not be used. There is no doubt but that if all chemists would take the same factor their results would agree. There is a difference between having results that agree and results that are correct. You can make the the most incorrect results agree, and you can make the most correct results disagree, if you change the subject on which you are working. So I would like to ask Mr. Gudeman and Mr. Leach, is it true that all glucose made now is subjected to the same degree of hydrolysis?

Mr. **Gudeman**. Mr. Wiley is correct in his statement. When I said that there was only one kind of glucose made I did not refer to the chemical composition of the glucose but was answering Mr. Leach's statement that there is one grade of glucose made for the jam manufacturer, another for the jelly manufacturer, and yet others for the brewer and the confectioner. The manufacturer makes only one grade of glucose and that is used by all the different manufacturers.

But this one grade of glucose will vary. I have shown in my article that the variation in the composition of glucose is very large, ranging, as regards the reducing power, from about forty to sixty. Mr. Wiley is correct; it is absolutely impossible for the manufacturer to duplicate the article. For practical purposes they are the same. But theoretically and chemically they are not the same.

The point I want to make is that the manufacturer only makes one grade of glucose to go on the market for the different submanufacturers. This glucose varies right along, and while theoretically the reducing power and the polarization have a certain ratio, and one can be calculated from the other, as a matter of fact it has never been found possible to control the manufacture of glucose by means of the polariscope, though it can be controlled by the iodin test.

Mr. **Leach**. I think the statement made by Mr. Gudeman that only one grade of glucose is manufactured might create a wrong impression. I do not think he means to say exactly that, because he knows that 45°, 43° and 42° glucose is sold to the trade—that is, there are three distinct grades. Of course, in one sense what Mr. Gudeman says is true, there is only one substance called glucose, but it is supplied to the trade in different grades. I happen to know that the New England Confectionery Association, which has absorbed all the large confectionery establishments in New England, uses only the 43° and 45° Bé. glucose. I also know that molasses mixers use only the 42° grade. Recently I visited the warehouse of the largest manufacturers of compound molasses in New England where, among 200 barrels of glucose all marked 42° Bé., I selected samples at random for analysis. The 45° Bé. glucose, although a liquid, is of such a consistency that you could turn a bottle of it upside down and it would not flow out for half an hour. It is so stiff that it can not be used in molasses, while the 42° Bé. glucose has just the right consistency for that purpose. That is, 45° Bé. glucose to be mixed with molasses must be watered down to the consistency of that product and then it virtually becomes 42° Bé. glucose. In the first place the 45° grade has a polarization much higher than 175, but when watered down the polarization decreases. So, I can not understand how there can be said to be only one grade of glucose when these three grades are made, all varying within certain limits, and are selected for use in the trade with special reference to their consistency.

Mr. **Gudeman**. On page 66 of your report of last year the subject is covered completely. When I say there is one grade of glucose, that is absolutely correct. You can figure on one grade and you can add as much water as you like and it does not change the glucose. In my original article I give there the extremes for the manufacture of glucose, ranging from 40 parts reducing substance to 60 parts of nonreducing substance against 55 parts of reducing substance and 45 parts of nonreducing substance. Then I go further and say—

> Glucose is a thick sirup, 41° to 45° Bé, containing 13 to 22.5 per cent of water. * * * If the conversion of starch with acid is carried to a point where a dilute iodin solution will just give a distinct color reaction, we have glucose. * * * The ratio of the reducing substance to nonreducing substance depends on the accuracy in stopping the conversion, for neutralization, at the exact point decided upon. This ratio determines whether the product is glucose or grape sugar, and no sharp dividing line exists. The rotating powers of glucose and grape sugar depend absolutely on this ratio. Actually no two batches of commercial glucose or grape sugar are identical; for all practical purposes they are alike, as a few points either way from the standard decided upon will make no difference in the appearance, taste, or working qualities of these products.

Now, that is the only difference. When you examine a product that contains the glucose it is absolutely impossible to tell what grade of glucose has been used, allowing that each percentage of water means a different grade. If the results are figured on the dry substance, as far as the commercial article is concerned, but one grade of glucose exists. The commercial article itself contains varying percentages of water. When a product is examined it is impossible to determine whether a grade of 41°, 43°, or 45° glucose has been used.

Mr. **Leach**. I only want to say one more thing and that is that each manufacturer uses the grade that is particularly adapted for his purpose. He is not going out of his way to change the material purchased by watering or boiling down. If he wants a thick glucose he buys the 45°, if he wants a thin glucose he buys 42°.

Mr. **Wiley**. There is just a little flaw in the cogent reasoning of Mr. Gudeman, and to illustrate it I will say that on the Board of Trade of Chicago, in which city Mr. Gudeman lives, there are three grades of wheat recognized in the market. I have no doubt that in each one of those three grades there are plenty of grains that belong to the other two grades, and yet the three grades are absolutely recognized, and I think we could not say that there is only one grade of wheat recognized on the Chicago market. They have distinct qualities, and each grade can be recognized by experts. So glucose has distinct grades, which can be recognized by the chemist, though the only variation be in the amount of water.

Mr. **Gudeman**. The German Government recognizes only one grade of glucose as having a certain specific gravity and consequently a certain percentage of water, and I only recognize one grade of glucose free from water, and base my remarks on that point. What Mr. Wiley says is true, but I would like to ask him how he can determine in a glucose mixture whether 41°, 42°, or 45° glucose has been used, and how he can differentiate them when mixed with molasses or any other product?

Mr. **Wiley**. It has been shown that the difficulty of incorporating a given grade of glucose in certain products is so great that it is not attempted, and therefore if we should find glucose in a certain mixture we would infer that it was not, let us say, 45° glucose.

Mr. **Leach**. I would like to make a recommendation that the method as it now stands be changed, to the effect that the results shall be expressed in terms of glucose polarizing at 175°, thus doing away with any uncertainty. I will frame such a recommendation and submit it to the committee on recommendations.

Mr. **Wiley**. I think that will be a step toward the solution, and it makes no difference whether the result be expressed as hydrous or as anhydrous glucose. It is a good suggestion to regard the solid substance only, and thus remove the difficulty.

Mr. **Leach**. Except that when we are called upon to express the quantity of commercial glucose in a mixture, in court, the statement as to the dry substance is not desired.

Mr. **Wiley**. It must be calculated to the commercial grade of water.

Mr. **Leach**. Yes; whereas if one determination is made and expressed as wet substance direct the whole ground is covered at once.

Mr. **Gudeman**. As I am not a member of this association, I could not make a motion, but I think the recommendation made by Mr. Leach covers the whole subject, and it agrees absolutely with my original suggestion that a constant factor be used rather than a variable one. I wish to express my thanks to the association for allowing me to take part in this discussion.

Mr. **Munson**. I would like to say a word or two in regard to the factor selected. In Mr. Gudeman's article in last year's proceedings the range in polarization of the dry material given for glucose is from 186° to 198°. The amount of water is stated to vary from 13 to 22.5 per cent. Assuming that the highest polarization of 198° is found with a glucose having the lowest percentage of water, and reducing the reading to the basis of the original glucose having 13 per cent of water, the highest polarization becomes only 172°, which bears out Mr. Leach's more recent work in which he has shown that the polarization of glucose is very much lower than formerly. It seems probable that the factor 175 is higher than that of any sample of glucose, even the 45° Bé., to be found in the trade.

Mr. **Leach**. It is true that later experiments seem to indicate that 42° Bé. glucose as now made polarizes lower than formerly. I selected 175 because that is the factor we have used for ten years, using the highest factor so as to give the manufacturer the benefit of the doubt. When asked in court how much glucose is present, we say, "at least" a certain per cent; there may be more. It is for this reason, and to avoid uncertainty, that I would recommend expressing results in terms of a certain factor. If it seems to be desirable to change this factor at any time it can be done.

The **President**. Committee C, of which Mr. Leach is chairman, has this subject under consideration, and any further material on the subject can be submitted to him.

The report on dairy products is in order.

Mr. **Cavanaugh** (referee on dairy products). It has been impossible for me to make a formal report this year. I recommend that the referee for next year take up the same subject, viz, the effect of preservatives on the albumin of milk with special reference to their quantitative determination.

Mr. **Patrick**. I wish to submit a note on the Waterhouse test for the detection of renovated butter, as modified at the Department of Agriculture.

NOTE ON THE WATERHOUSE TEST, MODIFIED, FOR THE DETECTION OF RENOVATED BUTTER.

By G. E. Patrick.

Two years ago I reported to the association the results of my work with this test as applied to the detection of renovated butter, and in view of certain experiences with the test since that time it seems advisable to make a further report.

In order to avoid confusion, it may be well to review the following facts: The Waterhouse test was originally proposed for the detection of oleomargarin,[a] and consisted in melting the butter or oleomargarin in hot milk (preferably skim milk) and stirring the mixture rapidly while it is cooled by placing the vessel in cold water or otherwise. The writer soon found that renovated butter would behave the same as oleomargarin upon the application of this test—that is, it would gather in a lump or single mass during the stirring unless the cooling was effected very rapidly, as by ice water. In order to make the test one for oleomargarin as distinguished from both renovated and genuine butter, and at the same time to make it useful to butter consumers who need such a test more than chemists do, household utensils were used and a practical way determined of obtaining the temperature, with those utensils, at which oleomargarin gathers in a lump, and butter, either genuine or renovated, granulates.[b] So much for the test as applied to the detection of oleomargarin.

Further, it was found that upon cooling much more slowly than is required in testing for oleomargarin, for example, with water at 12° C., instead of with ice water, renovated butter will gather in a lump or single mass, while, as a rule, genuine butter will granulate. The exceptions to the rule were thought to be butters that had been more or less melted, though possibly others were included, since a few butters not visibly damaged were among the exceptions. These results were obtained when testing with milk. Mr. Wiley suggested trying water instead of milk, and this worked so well that the former medium has been used in the greater part of our work ever since. The details of the test as conducted in this laboratory are as follows:

One hundred cubic centimeters of distilled water in a small tin cup, 2¾ inches in diameter, are heated to just below 50° C., a slightly rounded teaspoonful of the butter is added, and gently stirred until melted; the temperature is then raised to exactly 50°, the cup is immediately placed in a pan 9 to 9½ inches in diameter at the base, containing 1,000 cc of water at 12° C. The contents of the cup are then stirred rapidly with a wooden rod (more slender than a match) for 10 minutes with a circular and crosswise motion in turn, in such manner as to keep the fat well agitated. The water in the pan is thoroughly mixed once every minute, the cup being used as a stirrer. At the end of 10 minutes' stirring the test is finished.

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[a] J. Amer. Chem. Soc., 1901, p. 200.

[b] U. S. Dept. Agr., Farmers' Bul. No. 131.

In my report of two years ago it was stated, as the result of experience up to that time, that a butter which granulates in this test is above suspicion of having been renovated, and is surely genuine. Two years more of experience have shown that this view must be modified, for four samples of canned oleomargarin from Europe have been encountered that granulated like genuine butter in this test. Furthermore one sample of canned butter (No. 291 D. D.) also from Europe, which, while it was not a renovated butter judging from the microscopic examination, acted like one in the boiling test, but nevertheless granulated like a genuine butter in the modified Waterhouse test. The fat of this butter, examined after some months exposure, however, gave a Reichert Meissl number of 22.4 and a saponification number of 229.7, figures which certainly fail to prove adulteration. As to the four oleomargarins, it is probable that their behavior was caused by the kind of fats entering into their composition, since other European oleos opened at the same time behaved exactly as do American oleos—that is, they "gathered" in this test.

The failure of the test to detect oleomargarin is of little consequence to the chemist, since he has so many ready means of detecting an oleo. Failure to detect a renovated butter would be much more serious, but in the case cited it is an open question whether or not the test failed at all. The instances given above are the only ones yet encountered in which a butter that granulated in the test was not considered upon other grounds as being genuine, i. e., not renovated—and at least not grossly adulterated. What would be the effect of moderate adulteration in the churn, without melting, is an unanswered question.

Turning now to a consideration of the exceptions, it is found that it is as yet impossible to name with certainty all the causes for the behavior of the few genuine butters that gather in this test. From the first the writer has thought that partial melting of the butter by careless treatment while in transit or storage is one of the causes, but this is merely an assumption, as it has never been proved. Another cause recently discovered by the writer is of a similar nature and was observed in the following way: During the past year frequent analyses were made of butter from a dairy conducted on creamery methods, and it was found that this butter, although fresh and perfect in appearance, invariably gathered in a lump when subjected to the Waterhouse test, modified. This butter had two evident peculiarities; it was always without salt and was of a very mild flavor, so mild in fact that it was at once pronounced to be a sweet cream butter. That the absence of salt was not the cause of the abnormal behavior of the butter in this test was proved by repeating the trial after having salted the butter in the laboratory, the result being the same. For a long time the writer forced himself against reason to almost believe that the peculiar behavior of the butter was due to the fact that it was made, as was believed, from unripened cream, but finally the thought suggested itself that the butter might be made from pasteurized cream, and upon inquiry it was learned that about half of the cream used in the making of each lot of butter was pasteurized at 160° F. for 10 minutes, then mixed with the other half which was not pasteurized, and the whole ripened to an acidity of about 0.6 per cent, a fact which quite upset the theory that it was sweet cream butter.

Since these facts were ascertained the writer has tested samples of all the butters to be found in the Washington market which it was claimed were made from pasteurized cream. Only four brands were found, three in prints, the wrappers of which bore the statement that the butter was made from pasteurized cream; the fourth was tub butter, whose claim to pasteurization rested upon a verbal statement said to emanate from the manufacturer. Of these four samples only one gathered in the test; the other three granulated, thus placing themselves above suspicion. The question naturally arises as to whether these three butters were really made as alleged from pasteurized cream, and again whether, if so made, the temperature of pasteurization was not lower, or the time of its application shorter, than obtained in the manufacture of the samples which gathered in the test. For the answering of these questions there has as yet been neither time nor opportunity. In whatever way they may be answered in the future, or whatever facts may be developed, certainly those already observed indicate strongly that one of the causes which may cause a genuine butter to gather in the modified Waterhouse test is the pasteurizing of the cream from which it is made.

The PRESIDENT. If there are no other papers on this subject we will take up the report on nitrogen.

REPORT ON THE DETERMINATION OF NITROGEN.

By F. W. MORSE, *Referee.*

Eight laboratories cooperated in the work on available organic nitrogen. Some preliminary work on the neutral permanganate method was executed by Mr. L. A. Hill, of the New Hampshire station, and the department of foods and feeding of the Massachusetts station made some comparisons of various modifications of the alkaline method for determining availability.

As a result of these trials, the referee decided to use but two samples of materials, to leave the neutral method as it stood last year, and to try three modifications of the alkaline method. The preliminary work on the neutral method showed that marked variations in the effect of the permanganate solution on the nitrogenous substance were produced by changes in temperature, such as would be caused by a simmering water bath, or one boiling vigorously, and the use of hot solutions at the start instead of cold. This last modification produced such a marked effect on the solubility of the nitrogen, and increased so much the apparent availability, that it was deemed inadvisable to use it, but to emphasize the importance of a large water bath well filled and boiling vigorously.

The work with the alkaline permanganate solution showed the necessity of a fixed amount of distillate and the probable advantage of a larger volume of solution. The methods as sent to the various analysts were as follows:

The neutral permanganate method.—Into a 400 cc Griffin beaker, low form, weigh an amount of sample containing, approximately, 0.075 grams of nitrogen (samples containing material that has been treated with acid should be washed on a 9 cm S. S. No. 595 filter to 200 cc and transferred, filter and all, to beaker); digest this with 125 cc of permanganate of potash solution (16 grams of pure potassium permanganate in 1,000 cc of solution) in a steam or hot-water bath for thirty minutes. Have the beaker let down well into the steam or hot water and keep closed with a cover glass, stirring twice at intervals of ten minutes with a glass rod. At the expiration of the time remove from bath, add 100 cc of cold water, and filter through a heavy 15 cm folded filter. Wash with cold water, small quantities at a time, till total filtrate amounts to 400 cc. Dry and determine nitrogen in residue by Kjeldahl method.

The alkaline permanganate method.—Weigh out an amount of the sample containing, approximately, 0.045 gram of nitrogen, and transfer to a 600 cc distillation flask. After connecting with a condenser to which the receiver containing standard acid has been attached, digest with 100 cc of alkaline permanganate solution (16 grams of potassium permanganate and 150 grams of sodium hydrate in 1,000 cc of solution) for thirty minutes below the boiling point. Then boil slowly until the distillation is completed.

Since the samples contain superphosphate, it will be necessary to wash them as directed for the neutral method, but it is unnecessary for the alkaline method.

In the neutral method the time and temperature are important. The water bath should be well filled and boiling vigorously. If nearly empty, or barely boiling, it will take a longer time to bring the solution to its maximum temperature, and thus cause a marked variation in results. Filtering may be quickly done with the aid of a Hirsh funnel and filter pump.

Analysts are requested to try the following modification with the alkaline permanganate solution: Use 150 cc of solution, and after digesting 30 minutes distill off 100 cc. This method varies in results with varying amounts of distillate; consequently a uniform amount of distillate should produce more concordant results. Also try the same quantities of solution and distillate, omitting the thirty minutes digestion.

Dried blood and cotton-seed meal were again selected as types of organic materials to be used for testing the methods, since it was the referee's plan to get more concordant results from the different laboratories than were obtained last year. Both materials were sifted through a 0.5 mm sieve and mixed with an equally fine acid phosphate. The mixing was done as thoroughly as possible, but as the blood contained many hard granules it was expected that the different samples would vary somewhat in their content of total nitrogen, and this proved to be the case.

Nine analysts reported results on total nitrogen and eight on the various methods for the available. The results are given in the tables under the designations of the respective materials.

TABLE I.—*Total and available nitrogen—dried blood, sample No. 1.*

Analyst.	Total nitrogen.	Neutral method.		Alkaline method: Available nitrogen.		
		Insoluble nitrogen.	[a] Available nitrogen.	[b] 1.	[c] 2.	[d] 3.
T. C. Trescot, U. S. Department of Agriculture	[e] 4.20	2.06	2.19	2.40	3.01	3.01
	[f] 4.25	2.03		2.42	3.11	3.04
Department of foods and feeding, Hatch Experiment Station, Massachusetts	4.31	.54	3.78	2.66	2.90	....
		.52		2.70	....	....
				2.64	2.92	....
F. A. Urner, State Agricultural Experiment Station, New York	4.26	.93	3.41	2.64	2.87	2.91
		.94		....	....	....
		.67		2.65	2.97	2.77
A. B. Foster, Maryland Agricultural College	4.05	.68	3.37	3.04	2.84	2.32
				2.96	2.91	2.34
S. H. Sheib, Virginia-Carolina Chemical Co	[g] 4.29	.44	3.84	....	....	....
		.41		2.66	2.59	....
	[h] 4.28	.43		2.65	....	....
		.46		2.82	2.66	....
C. H. Jones, Agricultural Experiment Station, Vermont	4.35	.65	3.70	2.79	2.99	2.95
F. M. Hollister, Agricultural Experiment Station, Vermont	4.34	.48	3.86	2.80	2.98	2.94
L. A. Hill, Agricultural Experiment Station, New Hampshire	4.09	.50	3.59	2.65	3.26	3.31
		.49		2.59	3.37	3.32
W. H. Scherffius, Agricultural Experiment Station, Kentucky	[i] 4.27	.45	3.87	2.04	....	....
		.37		1.95	....	....
		.39		2.36	....	....
F. B. Carpenter, Virginia-Carolina Chemical Co	4.25	....	....	....	....	....

[a] Calculated by subtracting average of insoluble nitrogen from average total nitrogen.
[b] 100 cc solution, digested 30 minutes, distilled.
[c] 150 cc solution, digested 30 minutes, distilled 100 cc.
[d] 150 cc solution, no digestion, distilled 100 cc.
[e] Total by Gunning method; digested 4 hours.
[f] Digested 5 hours.
[g] Digested 1¼ hours.
[h] Digested 2 hours.
[i] Digested 5 hours.

TABLE II.—*Total and available nitrogen—cotton-seed meal, sample No. 2.*

Analyst.	Total nitrogen.	Neutral method.		Alkaline method: Available nitrogen.		
		Insoluble nitrogen.	[a]Available nitrogen.	[b]1.	[c]2.	[d]3.
T. C. Trescot, U. S. Department of Agriculture	[e]3.81 [f]3.81	.47 .47	3.34	1.52 1.52	1.89 1.92 1.89	2.01 1.96
Department of foods and feeding, Hatch Experiment Station, Massachusetts	3.83	.25 .24 .24	3.59	1.63 1.62	1.88 1.86	....
F. A. Urner, State Agricultural Experiment Station, New York	3.85	.48 .59 .50	3.33	1.89 1.82	2.25 2.34	2.19 1.96
A. B. Foster, Maryland Agricultural College	3.89	.48	3.41	1.77 1.86	2.38 2.47	1.37 1.37
S. H. Sheib, Virginia-Carolina Chemical Co	[g]3.80 [h]3.77	.21 .24 .24 .22	3.57	1.67 2.14 1.87 2.09 1.76	2.36 2.42 2.43 2.18 2.51	....
C. H. Jones, Agricultural Experiment Station, Vermont	3.86	.32	3.54	1.74	2.47	2.29
F. M. Hollister, Agricultural Experiment Station, Vermont	3.91	.29	3.62	1.70	2.35	2.38
L. A. Hill, Agricultural Experiment Station, New Hampshire	3.84	.19 .19	3.65	1.75 1.75	2.25 2.26	2.27 2.30
W. H. Scherffius, Agricultural Experiment Station, Kentucky	[i]3.84	.16 .25 .21	3.63	1.30 1.44 1.26	....	....
F. B. Carpenter, Virginia-Carolina Chemical Co	3.81	....	....	....	....	....

[a] Calculated by subtracting average of insoluble nitrogen from average total nitrogen.
[b] 100 cc solution, digested 30 minutes, distilled.
[c] 150 cc solution, digested 30 minutes, distilled 100 cc.
[d] 150 cc solution, no digestion, distilled 100 cc.
[e] Total nitrogen by Gunning method; digested 4 hours.
[f] Digested 5 hours.
[g] Digested 1½ hours.
[h] Digested 2 hours.
[i] Total digested 5 hours by Kjeldahl method.

The parallel determinations of total nitrogen by the individual analysts were in all cases very close and were, therefore, averaged in the table. The average results of eight analysts using the neutral method, compared with those of eight analysts using the first and second modifications of the alkaline method, are as follows:

TABLE III.—*Total nitrogen recovered as available.*

Method.	Blood.	Cotton-seed meal.
	Per cent.	*Per cent.*
Neutral	86.5	91.6
Alkaline (1)	63.4	46.1
Alkaline (2)	69.4	58.1

Several of the analysts tried other modifications. Four made comparisons of digested and undigested charges using 100 cc of the alkaline solution. The digested samples were heated for 30 minutes before distilling, and the undigested ones were distilled without the preliminary heating. Their results follow:

TABLE IV.—*Available nitrogen recovered by modified alkaline method.*

Analyst.	No. 1, blood.		No. 2, cotton seed.	
	Digested.	Undigested.	Digested.	Undigested.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
F. A. Urner	2.64	2.84	1.89	1.93
	2.65	2.93	1.82	1.94
C. H. Jones	2.79	2.68	1.74	1.68
F. M. Hollister	2.80	2.60	1.70	1.64
A. B. Foster	3.04	2.92	1.77	1.42
	2.96	2.88	1.86	1.60

Mr. Foster also tried the following modification: Use 150 cc alkaline solution, digest 30 minutes and distill 100 cc, running in water through a tap funnel at the same rate as the distillate comes over, thus keeping the volume of liquid in the flask nearly constant. The available nitrogen recovered in sample No. 1, blood, was 2.55, 2.56; in No. 2, cotton seed, 1.68, 1.69.

The department of foods and feeding of the Massachusetts experiment station reported results with the use of 200 cc of alkaline solution, digestion for one hour and distillation of 100 cc. The available nitrogen recovered in sample No. 1, blood, was 2.88 per cent, 3.06 per cent; in No. 2, cotton seed, 2.33 per cent, 2.32 per cent.

Mr. Sheib sent some interesting results showing the variations due to different amounts of distillate and solution in the alkaline method. His results are given below:

TABLE V.—*Results obtained by varying amounts of distillate and solution in alkaline method.*

Sample No. 1.			Sample No. 2.		
Quantity of solution employed.	Final volume of distillate.[a]	Available nitrogen.	Quantity of solution employed.	Final volume of distillate.[a]	Available nitrogen.
cc	*cc*	*Per cent.*	*cc*	*cc*	*Per cent.*
200	190	2.67	250	225	2.86
200	190	2.91	200	185	2.96
150	140	2.89	200	190	2.93
150	100	2.66	150	100	2.42
150	110	2.90	150	100	2.36
150	100	2.59	150	100	2.43
150	145	3.11	150	100	2.18
150	140	3.06	150	100	2.51
100	95	2.82	150	130	2.47
100	90	2.65	100	95	1.67
100	90	2.66	100	95	2.14
			100	95	1.87
			100	95	2.09
			100	90	1.76

[a] Approximate.

The following notes were made from the letters of the different analysts:

Department of Foods and Feeding, Amherst, Mass.—Attention is again called to the use of sulphate of potash in the plain Kjeldahl method, as it insures more perfect and more rapid oxidation. It increases the percentage of nitrogen slightly and decreases the time of digestion to relative clearness about one-half. In the neutral permanganate method the filtrate from No. 1 shows a slight excess of permanganate and none in No. 2. The immediate distillation with the alkaline permanganate was not tried, as too much time is required for the reaction.

A. B. Foster, Maryland.—In the alkaline permanganate method using 100 cc and distilling directly until as much as possible had distilled over, the flasks containing the cotton-seed meal were found to froth badly. The last 5 cc that came over was stronger than centi-normal ammonia solution. It was also observed that it made considerable difference whether the distillation took place rapidly or slowly; the more slowly it was boiled the more ammonia distilled over.

C. H. Jones, Vermont.—The total nitrogen charges were digested from two and one-half to three hours. In the alkaline permanganate method, when the preliminary digestion is omitted, frothing is apt to occur. This is not so troublesome with the samples here reported as with garbage tankage and other low-grade materials.

F. A. Urner, New York.—For total nitrogen 1 gram was taken as a charge. It was allowed to stand over night with 20 cc sulphuric acid. In the morning it was digested slowly for one hour and then boiled for one and one-half hours. With the alkaline permanganate method, in both samples when only 100 cc of solution were used, results were low, and these were thought to be due to the fact that only 75 cc of distillate could be recovered.

C. D. Howard, associate chemist of the West Virginia station, in a communication gave the following experience with the neutral method for available nitrogen in the practical work of the fertilizer control: With ordinary medium grade fertilizers the use of a charge equivalent to 0.075 gram of nitrogen did not seem uniformly practicable, as the quantity of material represented is usually considerably more than sufficient to exhaust the 125 cc of permanganate solution. Consequently in all mixed goods a uniform charge of 2 grams was used. It was found that in this way much more concordant results could be obtained. The majority of brands met with at that station are made up on a guarantee of 0.82 per cent of nitrogen. According to the method as laid down, it would require over 9 grams for a charge, an amount which would include more organic matter in itself than would be required to exhaust the permanganate.

RECOMMENDATIONS.

From the data and observations obtained this year, I recommend that the first part of the neutral method be changed to read as follows: Into a 300 cc low-form Griffin beaker weigh 2 grams of material, if the sample is a mixed fertilizer; if it is from concentrated goods, use an amount containing approximately 0.075 gram of nitrogen.

I also recommend that the study of methods for the determination of available organic nitrogen be continued.

Mr. **CARPENTER.** I have a paper on this subject prepared by Mr. Sheib to present to the association:

THE ELECTROLYTIC REDUCTION OF NITROGEN IN METALLIC NITRATES.

By S. H. SHEIB.

William H. Easton, in the Journal of the American Chemical Society,[a] describes "The reduction of nitric acid in metallic nitrates to ammonia by the electric current." The results published, however, vary on a sample of chemically pure potassium nitrate between 13.32 per cent and 13.77 per cent, theory requiring 13.86 per cent.

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[a] Vol. 25, No. 10, October, 1903.

In the analysis of potassium nitrate Easton employed 0.5 gram each of potassium nitrate and copper sulphate under the following conditions: Cathode, platinum or copper plate, 100 sq. cm area; ampères, 0.15 to 3; volts, 3 to 8; dilution, 150 cc; sulphuric acid (sp. gr. 1.062), 30 cc; time, for 3 ampères, 1¼ hours; for 0.15 ampère, 8½ hours. The solution was electrolyzed until the copper was completely deposited, after which the ammonia was determined by distillation in the usual manner.

Owing to the many objections to existing methods the electrolytic reduction as described by Easton was tried, but the results were far from satisfactory. A dried sample of sodium nitrate chemically pure, theoretically containing 16.47 per cent of nitrogen, yielded from 15.96 per cent to 16.22 per cent. A sample of potassium nitrate similarly dried gave from 13.38 per cent to 13.68 per cent (theory 13.86 per cent). These results agree fairly well with those obtained by Easton, but are still decidedly unsatisfactory. The liquid after reduction of all the copper gave, in most instances, a decided reaction with brucine, and it was hence deemed advisable to increase the proportion of copper sulphate. Further, since the odor of nitric acid was perceptible during the progress of the reduction, particularly at the start, the quantity of sulphuric acid (30 cc, sp. gr. 1.062) was reduced to 20 cc. It would seem that this quantity might be still further reduced without danger of loss of ammonia, or without materially affecting the conductivity of the solution, since the acid set free by the decomposition of the copper sulphate continually replaces the portion combining with the reduced ammonia.

Easton says: "In no trial did the variation of the current density at the anode have any influence on the amount of nitrogen reduced to ammonia." The experiments of the writer have not borne this out, but have rather tended to confirm the statement of Vortmann: "It is advantageous to use a feeble current (1 to 2 cc oxyhydrogen gas per minute)."

With currents of 1 ampère or over, the results were invariably low, the error increasing with the strength of the current, while the employment of currents of 0.3 ampère or less, gave results that were uniform, and approached theory very closely. In the examples given below, the following conditions were observed:

Temperature, that of room;
Volume of solution, 200 to 250 cc;
0.35 to 0.5 gram sodium nitrate;
1 to 2 grams copper sulphate;
20 cc sulphuric acid, sp. gr. 1.062;
Cathodes, platinum cylinders, 100 sq. cm area;
Current density, 0.15 to 0.30 ampère;
Potential, 2 to 3 volts.

The reduction was allowed to proceed over night, and the distillation and titration were performed in the usual manner.

The presence of spongy copper on the cathode seemed to exert an influence rather favorable than otherwise. While it is true that portions of the spongy deposit became detached, no effort was made to remove them from the liquid, which was distilled as usual.

Results of electrolytic reduction of nitrogen in three samples of nitrate of soda.

C. P. NITRATE OF SODA, DRIED AT 130°.

	Nitrogen.	Ammonia.	Sodium nitrate.
	Per cent.	*Per cent.*	*Per cent.*
Sample 1	16.44	19.96	99.79
Sample 2	16.48	20.02	100.01
Sample 3	16.46	19.98	99.91
Average ..	16.46	19.98	99.91
COMMERCIAL SAMPLE NITRATE OF SODA, NO. 43496.			
Sample 1	15.76	19.14	95.66
Sample 2	15.80	19.18	95.91
Average ..	15.78	19.16	[a] 95.78
COMMERCIAL SAMPLE NITRATE OF SODA, NO. 43698.			
Sample 1	15.88	19.28	96.39
Sample 2	15.84	19.22	96.15
Sample 3	15.88	19.28	96.39
Average ..	15.86	19.26	96.31

[a] By method of difference, 96.03 per cent sodium nitrate.

Mr. Hopkins. I would like to ask the referee if either of these methods for the determination of available nitrogen has been applied to materials which are pretty well known in practice to be readily available and to others not equally available, such as leather and dried blood, for example; have comparisons been made with those materials?

Mr. Morse. These comparisons have been made by previous referees; and as regards the effect on materials, the neutral method was found to agree with simultaneous vegetation experiments which the referees carried on and with vegetation experiments conducted at the Connecticut Agricultural Experiment Station.

As to the alkaline method, no one claims anything for it except that it sorts out the poor nitrogenous material from the good, but vegetable nitrogenous material, such as cotton-seed meal, always gives low results. Therefore the trouble with the alkaline method is that it needs a check on any low result. It is claimed, however, by those who are using it right along in the routine work of fertilizer analysis that they can rapidly sort out the good nitrogen from the bad and then check the bad with some other method, so that the better class of the vegetable nitrogenous material can be distinguished.

Mr. Huston. I have noted with some interest the comparison or analogy drawn between the question of taking a fixed quantity of the sample, an arbitrary fixed quantity, and our practice in connection with insoluble phosphoric acid. I suppose that quantity in connection with insoluble phosphoric acid is so fixed now in practice that it will be difficult and perhaps impossible to change it. Yet, with two or three products the method for insoluble phosphoric acid is not right at all, simply because these products contain from 35 to 50 per cent of citrate soluble phosphoric acid, and the solvent power of 100 cc of the official citrate solution is not great enough to hold up that much phosphoric acid. I thought it might be well to mention in this connection that you might get into the same sort of trouble if you adopt for the nitrogen work a single fixed quantity. It seems to me that you will be on the safe side if you have a somewhat flexible quantity, and it is easier to consider the point now than it will be after you have adopted a fixed quantity.

The case seems not unlike that of basic slag, in which the free lime in a slag neutralizes a considerable quantity of citric acid before any citric acid is available for dissolving the phosphate. So possibly some of the weak acids, or other organic matter, are really using up the permanganate solution before it gets a chance at the nitrogenous products, and just as a definite quantity of citric acid is allowed for free lime in slag so allowance may be made for this nonnitrogenous matter irrespective of the nitrogen contained in the sample.

Mr. MORSE. I tried to provide in a measure for just that point. As Mr. Huston has said, we recognize that we have had much difficulty with the ammonium-citrate method and have tried to obviate that difficulty in mixed fertilizers and materials by using a fixed amount of nitrogen in the latter case and a fixed quantity of sample in the former.

The PRESIDENT. The next subject is the separation of nitrogenous bodies.

Mr. VAN SLYKE. A letter from Mr. Harcourt states that owing to illness and death in his family he has not been able to put his results on vegetable proteids together for publication and he suggests that he be allowed to finish his report, which by the way is a compilation, and forward it later for publication. This can be acted upon later.

The subject of my own work was the separation of nitrogenous bodies in dairy products.

REPORT ON SEPARATION OF NITROGENOUS BODIES IN MILK AND CHEESE.

By L. L. VAN SLYKE, *Referee.*

In the referee's report last year the writer described, among other methods, one devised by Mr. E. B. Hart and himself for the determination of unsaturated paracasein lactate, that is, paracasein monolactate, in cheese. Since in normal American cheddar cheese we have at the start only paracasein and paracasein monolactate, the separation of these compounds is readily affected by the method given; that is, by dissolving the paracasein monolactate in a 5 per cent solution of sodium chlorid. Under rare and abnormal conditions we may possibly have a mixture of paracasein, paracasein monolactate, and paracasein dilactate. Since we have not yet been compelled to face a condition of this kind, no provision has been made for such an emergency, and at present no satisfactory method can be given for the separation of paracasein and paracasein dilactate.

Mr. Hart and I have been giving our attention recently to the study of the corresponding casein salts, casein monolactate and casein dilactate, and their formation in the ordinary souring of milk has been studied. Some study has been made also of the relation of these compounds to the determination of casein in milk. While this work is not yet completed, it is deemed desirable to report progress at this time.

I. CASEIN MONOLACTATE AND CASEIN DILACTATE.

1. *Determination of casein monolactate in milk.*

Casein monolactate in milk coagulates readily at 40° C. Hence, in a milk containing only casein and casein monolactate, the monolactate can be separated by heating the milk to about 40° C., filtering the precipitate found, washing, and determining the nitrogen in the precipitate. Ten grams of milk diluted with 90 cc of water give good results.

2. *Separation of casein monolactate and casein dilactate.*

Casein dilactate coagulates completely at 40° C. and below. In milk containing casein monolactate and dilactate, heat 10 grams of milk diluted with 90 cc of water to 40° C., and these two salts precipitate and are separated from the milk casein by filtration. The washed precipitate is then treated with 100 cc of a 5 per cent solution of sodium chlorid and the whole heated to 55° C. with frequent agitation for two hours. The process is facilitated somewhat by the presence of pure quartz sand. The casein monolactate goes into solution and is separated from the casein dilactate by filtration and washing.

3. *Separation and determination of casein; casein monolactate and casein dilactate in milk.*

(*a*) The total amount of nitrogen precipitated by acid is determined by the official method prescribed for determining casein in milk.

(*b*) Heat 10 grams of milk diluted with 90 cc of water to 40° C. for fifteen or twenty minutes, filter the precipitate formed, and wash with distilled water. The precipitate is then transferred to a small Erlenmeyer flask provided with a stopper, treated with 100 cc of a 5 per cent solution of sodium chlorid, and heated at 55° C. for two hours, with frequent agitation. The mixture is then filtered, the remaining precipitate washed with water, and the nitrogen determined in both the precipitate and filtrate. The nitrogen in the precipitate represents casein dilactate; that in the filtrate, casein monolactate. The sum of these two subtracted from the total nitrogen found by precipitation with acid gives the amount of nitrogen as casein.

II. THE OFFICIAL METHOD FOR DETERMINATION OF CASEIN IN COW'S MILK.

Ten years ago, when I first submitted to this association the present official method for the determination of casein in cow's milk, the question suggested itself as to what the precipitating action of acid was upon casein and whether there was a chemical union of acid and casein. In the light of the recent discoveries referred to above, this query may now be answered. When 10 grams of cow's milk is treated with 1.5 cc of 10 per cent acetic acid, the precipitate formed is wholly casein diacetate. Some results of our work, not yet completed, show that much less acid than this is required to form the diacetate. It is probable that we shall find it desirable to reduce the amount of acetic acid used to 1 cc or less.

In this connection a fact to which the writer called attention some years ago can now be explained. In the case of most milks it was found that if the filtrate from the acetic acid precipitate of casein was neutralized, we obtained more or less precipitate, usually about one-tenth of 1 per cent of casein. The suggestion offered was that this neutralization precipitate was due to solution of the casein precipitate in excess of acid. We now know that this explanation is true, and why. Saturated salts formed by casein and acids dissolve quite easily in excess of free acid. With the interpretation we give of the action of acids upon casein, there is good reason to believe that our method can be so modified as to avoid this error and also avoid the additional determination of a neutralization precipitate. Another year we shall be able to report the results of a completed investigation in this line.

III. Relation of Casein Monolactate and Casein Dilactate to the Hess and Doolittle Method of Detecting "Renovated" Butter.

This test is based upon the difference alleged to exist between the characteristics of the curd derived from pure butter and that derived from renovated butter. According to the statement of Hess and Doolittle, "the curd from true butter will have an amorphous, nongranular appearance, while the curd from "process" butter has a very coarse curdy appearance. The one is the proteid of cream; the other is the proteid of milk; the one is a gelatinous, ropy mass; the other is a granular, easily divided substance." In respect to chemical characteristics, Hess and Doolittle state that the fat-free curd and brine obtained from genuine butter give only a slight milkiness when treated with acetic acid, while the filtrate from "renovated" butter gives an abundant white precipitate. Mr. Patrick presented an exhaustive study of this test at our meeting two years ago. He spoke of the proteid of butter serum which coagulates on addition of acetic acid as "soluble casein," and found that the test did not suffice to distinguish between genuine butter and "process" butter.

Upon considering the process of butter making, it is readily understood that in the course of cream ripening lactic acid is formed and this unites with casein. If the ripening is slight—that is, if only a moderate amount of acid is developed, only casein monolactate is formed; if the cream is allowed to become sourer, a mixture of casein monolactate and dilactate or only dilactate is formed. Suppose only casein monolactate is formed, then the curd in the butter is casein monolactate. When such butter is melted the salt dissolves and with the casein monolactate falls to the bottom of the vessel. The salt brine dissolves the casein monolactate, which readily precipitates on addition of acetic acid. Moreover, the description of curd given by Hess and Doolittle as a gelatinous, ropy mass applies exactly to the brine-dissolved casein monolactate. What they describe as a "granular, easily divided substance," is very clearly casein dilactate. It should be stated that frequently, even when the casein in butter is present as dilactate, the curd suspended in the brine appears to be in a very finely divided condition, caused undoubtedly by the agitation produced in churning. Another point is that casein dilactate appears to be somewhat soluble in salt brine of a concentration such as we find in the serum of salted butter. Under these circumstances, if the butter serum is filtered the filtrate usually will give a precipitate on the addition of acetic acid.

Another point of interest stated by Mr. Patrick is the gradual disappearance of "soluble casein" in butter. In other words, fresh butter containing only casein monolactate may form enough acid from the occluded milk sugar to convert the casein monolactate into casein dilactate. Prolonged action of salt on the proteid of the butter in many instances may also account for the presence of coarser particles of curd in older butters.

It can therefore readily be seen, in the light of the existence of two such compounds as casein monolactate and casein dilactate, that it is a mere question of the amount of acid formed in the ripening of cream or milk as to whether one set of characteristics or another will be present. We can make genuine butter that will respond to the Hess and Doolittle test for "process" butter, or vice versa. Moreover, where a fresh, genuine butter responds to the test for pure butter, the same butter, if kept some weeks, responds to the test for a "process" butter. Such a test must be regarded as unreliable, and there does not at present seem to be much hope of finding a method of distinguishing between genuine butter and "process" butter on the principle proposed by the Hess and Doolittle test.

RECOMMENDATIONS.

Your referee suggests the following recommendations:

1. That the method given for the separation and determination of casein monolactate and casein dilactate be adopted as a provisional method.
2. That Mr. Harcourt's report on vegetable proteids be included in the printed proceedings.

REPORT ON THE SEPARATION OF VEGETABLE PROTEIDS.

By R. HARCOURT, *Associate Referee.*

Your referee found it impossible to do any extended investigation work on the separation of the vegetable proteid bodies and has confined his attention more to reviewing the literature on this subject. This report is, therefore, largely a compilation of the results obtained by those who have given this matter particular study. Many quotations are from the findings of Osborne and his coworkers.

Under the generic name of proteids are classed a number of highly complex compounds. As a class they are white or yellowish nonvolatile solids, usually, but not invariably, amorphous. They are unchangeable in the dry condition, but putrify readily when moist or in solution. Some of the proteids are soluble in water, others in neutral salt solutions, and some in alcohol, while they appear to be invariably insoluble in ether, chloroform, and carbon disulphid. They are mostly precipitated from their solutions by a great number of reagents, including certain salts of the light metals, salts and hydroxids of the heavy metals, mineral acids, certain organic acids, etc. According to Osborne,[a] the proteids are basic bodies and enter into some reactions with acids, with which they form true salts. They are decomposed by treatment with strong mineral acids, ammonia, leucin, tyrosin, and various other bodies being products of the decomposition. The amid compounds, of which asparagin and leucin are types, coexist in the plant with the higher proteids, and are probably intermediate compounds in the formation of the proteids. They occur in all living matter, being essential constituents of the protoplasm.

The various proteids differ somewhat in elementary composition. Hoppe-Seyler[b] gives the following limits:

Limits in composition of proteids (Hoppe-Seyler).

Data.	Carbon.	Hydrogen.	Nitrogen.	Sulphur.	Oxygen.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Minimum	51.5	6.9	15.2	0.3	20.9
Maximum	54.5	7.3	17.0	2.0	23.5

Osborne's analyses of at least two globulins, legumin, and edestin, indicate that the limit for nitrogen is not wide enough, for both these substances contained over 18 per cent of nitrogen.

While the proteids do not vary widely in composition, there is sufficient difference in their general properties to allow them to be arranged in groups.

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[a] J. Amer. Chem. Soc., **24**: 76.

[b] Watt's Dictionary of Chemistry, **4**: 327.

(*a*) *Vegetable albumens* have been found in the cereal grains. They are soluble in water, and coagulate at 70°. Sodium chlorid, magnesium sulphate, and acetic acid fail to precipitate them from their solutions.

(*b*) *Vegetable globulins* appear to be of very wide occurrence. They form the greater part of the proteids of the grains of the legumes and are found in smaller quantities in the cereal. They are insoluble in pure water and soluble in sodium chlorid solutions, from which they are precipitated by separating the salt from their solutions by dialysis. Coagulation takes place when their solutions are heated to 55° to 75°.

(*c*) *Vegetable albuminates.* It is now thought that the albuminates do not exist as such in plants, but that they are produced by the action of the reagents employed in the separation of the albumens and globulins. The researches of Hoppe-Seyler, Vines, Green, Martin, Osborne, and others have shown that the legumin and conglutin separated by Braconnot and Ritthausen are not albuminates, but true globulins. Osborne and Campbell[a] have shown that minute quantities of acid materially affect the solubility of globulins in salt solutions.

(*d*) *Vegetable proteoses* are found in the aleurone grains. These substances are intermediate in the formation of peptones from the higher proteids. They differ very slightly from the peptones, being, like them, soluble and slightly diffusible.

(*e*) *Vegetable peptones.*—True peptones apparently do not exist as such in the plant except transiently. They are probably formed by the action of ferments upon the proteids, and as peptones are very soluble in water and diffusible, they may thus pass to the growing portion of the organism.

(*f*) *Insoluble vegetable proteids* have an undoubted existence, in the sense that they can not be dissolved out of the vegetable tissues by solvents not acting chemically on them; but it is probable that such insoluble proteids do not preexist in the plant, but are produced by the action of ferments similar to those which occasion the coagulation of blood and milk. "The most important number of the group is gluten, a tenacious, tasteless, brownish-gray substance. It consists of two substances, one soluble in alcohol, which has been called 'gliadin' and another portion insoluble in alcohol, but soluble in weak alkali, called 'glutenin'".[b] In contradiction to this Osborne[c] states: "No ferment action occurs in the formation of gluten, for its constituents are found in the flour having the same properties and composition as in the gluten."

A vast amount of work has been done on the separation of these various classes and of individual proteid substances. Newer and more delicate methods of analysis have greatly aided this work and have exposed many of the errors of the earlier investigations. In this report I will not dwell on the conclusions of the earlier investigators, but shall seek to give the results of the latest work on some of the more important food products.

PROTEIDS OF WHEAT.

Leibig, Boussingault, Dumas, De Saussure, and many others have worked on the proteids of wheat. The latest and most complete work is reported by Osborne and Voorhees.[d] According to these investigators there are five proteids in wheat. The following is their summing up of the investigations of the wheat kernel:

The proteids contained in the wheat kernel are:

I. A globulin belonging to the class of vegetable vitellins, soluble in saline solutions, precipitated therefrom by dilution and also saturation with magnesium sulphate or ammonia sulphate, but not by saturation with sodium chlorid. Partly precipitated by boiling, but not coagulate at temperatures below 100°. The wheat kernel contains between 0.6 and 0.7 per cent of this globulin. When dried at 110° its composition was found to be as follows:

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[a] J. Amer. Chem. Soc., **19**: 482.

[b] Ingle, Manual of Agricultural Chemistry, p. 214.

[c] Amer. Chem. J., **15**: 471.

[d] Amer. Chem. J., **15**: 392.

Wheat globulin.

	Per cent.
Carbon	51.03
Hydrogen	6.85
Nitrogen	18.39
Sulphur	.69
Oxygen	23.04
	100.00

II. An albumin, coagulating at 52°, which differs from animal albumin in being precipitated on saturating its solutions with sodium chlorid or with magnesium sulphate, but not precipitated on completely removing salts by dialysis in distilled water. It was found to form between 0.3 and 0.4 per cent of the wheat kernel, and to have the following composition when separated from solution in the coagulated form by heating to 60° C.:

Wheat albumin.

	Per cent.
Carbon	53.02
Hydrogen	6.84
Nitrogen	16.80
Sulphur	1.28
Oxygen	22.06
	100.00

III. A proteose, precipitated (after removing the globulin by dialysis and the albumin by coagulation) by saturating the solution with sodium chlorid, or by adding 20 per cent of sodium chlorid and acidifying with acetic acid. This body was not analysed in its unaltered form. On concentrating its solutions by boiling, a coagulum was gradually developed which formed about 0.3 per cent of the wheat kernel and had the following composition:

Coagulum.

	Per cent.
Carbon	51.86
Hydrogen	6.82
Nitrogen	17.32
Sulphur Oxygen	24.00
	100.00

The solution filtered from the substance just described still contained a proteose-like body which was not obtainable in a pure state. Its amount could only be roughly estimated by precipitating the concentrated filtrate from the preceding substance with alcohol and multiplying the nitrogen contained in the precipitate by 6.25. The amount of this proteose was from 0.2 to 0.4 per cent of the seed. Both these substances are unquestionably derivatives of some other proteid in the seed, presumably the proteose first mentioned.

IV. Gliadin, soluble in dilute alcohol and forming about 4.25 per cent of the seed. It has the following composition:

Gliadin.

	Per cent.
Carbon	52.72
Hydrogen	6.86
Nitrogen	17.66
Sulphur	1.14
Oxygen	21.62
	100.00

This is the proteid called gliadin by Taddei and plant gelatin by Dumas and Cahours. Mixed with impurities or altered to a greater or less extent by the process of separation, it has been described by Ritthausen under the name of gluten fibrin, plant gelatin, or gliadin, and mucedin, and by Martin has been termed insoluble phytalbumose. The mucin of Berzelius and of De Saussure were impure preparations consisting chiefly of this proteid. It is soluble in distilled water to opalescent solutions which are precipitated by adding a little sodium chlorid. It is completely insoluble in absolute alcohol, but slightly soluble in 90 per cent alcohol, and very soluble in 70 to 80 per cent alcohol and is precipitated from these solutions on adding either much water or strong alcohol, especially in the presence of much salts; soluble in very dilute acids and alkalies, and precipitated from these solutions by neutralization, unchanged in properties and composition. This proteid is one on which the formation of gluten largely depends.

V. Glutenin, a proteid insoluble in water, saline solutions and dilute alcohol, which forms the remainder of the proteids of the wheat kernel, generally about 4 to 4.5 per cent of the seed. This substance is soluble in dilute acids and alkalies and is precipitated from such solutions by neutralization. Dissolved in 0.2 per cent potash water, precipitated by neutralization and, after thorough extraction with alcohol and either, again dissolved in potash water, the solution filtered clear, precipitated by neutralization and dried at 110°, it has the following composition:

Glutenin.

	Per cent.
Carbon	52. 34
Hydrogen	6. 83
Nitrogen	17. 49
Sulphur	1. 08
Oxygen	22. 26
	100. 00

Unless prepared as above described, the impurities are not removed and the analyses are discordant. This proteid was first described by Taddei under the name of zymon. Liebig as well as Dumas and Cahours named it plant fibrin. Ritthausen called it gluten casein, Weyl and Bischoff considered it to be an albuminate form of a myosin-like globulin. Martin named it gluten fibrin and likewise considered it to be an albuminate form of a myosin-like globulin.

VI. Wheat gluten is composed of gliadin and glutenin. Both these proteids are necessary for its formation. The gliadin with water forms a sticky medium, which by the presence of salts is prevented from becoming wholly soluble. This medium binds together the particles of flour, rendering the dough and gluten tough and coherent. The glutenin imparts solidity to the gluten, evidently forming a nucleus to which the gliadin adheres and from which it is consequently not washed away by water. Gliadin and starch mixed in the proportion of 1:10 form a dough, but yield no gluten, the gliadin being washed away with the starch. The flour freed from gliadin gives no gluten, there being no binding material to hold the particles together so that they may be brought into a coherent mass.

Soluble salts are also necessary in forming gluten, as in distilled water gliadin is readily soluble. In water containing salts it forms a very viscid, semifluid mass, which has great power to bind together the particles of flour. The mineral constituents of the seeds are sufficient to accomplish this purpose, for gluten can be obtained by washing a dough with distilled water.

VII. No ferment action occurs in the formation of gluten; for its constituents are found in the flour, having the same properties and composition as in the gluten, even under those conditions which would be supposed completely to remove antecedent proteids or to prevent ferment action. All the phenomena which have been attributed to ferment action are explained by the properties of the proteids themselves as they exist in the seed and in the gluten.

PROTEIDS OF RYE.

The proteids of this seed have not been as exhaustively studied as those of the wheat. Einhof, Heldt, Ritthausen, and Von Bibra succeeded in separating proteids similar to the gliadin and glutenin of wheat. Verdeil[a] claimed to have washed gluten out of rye flour, but did not consider it pure because it was contained with a substance soluble in alcohol. The following are the conclusions arrived at by Osborne after his study of the rye kernel:[a]

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[a] Ann. d. Chem. u. Pharm., 58: 319.

> Owing to the gum already mentioned the filtration and treatment of the rye extracts was difficult and prolonged and the amounts of globulin, albumin, and proteose could not be determined separately, as in the case of wheat. The rye flour contained 1.52 per cent of nitrogen. If we assume that the proteids of rye contain on the average 17.6 per cent of nitrogen, as was very nearly the case with those of wheat, and that all the nitrogen exists in proteid form, this sample of flour would contain 8.63 per cent of proteid. We have, therefore, 2.44 per cent of insoluble proteid and 6.19 per cent soluble in salt solution and alcohol.
>
> We have already shown that the alcohol-soluble gliadin amounted to 4 per cent of the flour and the leucosin to 0.43 per cent. There thus remains 1.76 per cent to be divided between edestin and proteoses.

	Per cent.
Insoluble in salt solution	2.44
Gliadin, soluble in alcohol	4.00
Leucosin, soluble in water	0.43
Edestin and proteose, soluble in salt solution	1.76
	8.63

PROTEIDS OF BARLEY.

Even less attention has been paid to the study of the proteids of barley than to those of rye. The conclusions reached by Osborne after an exhaustive study of the proteids of barley are as follows:[b]

> The barley kernel contains—
>
> I. Leucosin coagulating at 52°, which is the same as the albumin found in the wheat and rye kernels. Its composition, as shown by the average of six analyses, is:

	Per cent.
Carbon	52.81
Hydrogen	6.78
Nitrogen	16.62
Sulphur	1.47
Oxygen	22.32
	100.00

> This substance forms about 0.3 per cent of the seed.
>
> II. A small quantity of proteose, the reactions and composition of which could not be definitely ascertained.
>
> III. Edestin, a globulin which is the same as that found in the wheat and rye kernels and in a large number of other seeds. Its composition is approximately shown by the figures given below. Owing to the small amount of this body and the difficulty in preparing it, no perfectly pure preparations were obtained.

	Per cent.
Carbon	50.88
Hydrogen	6.65
Nitrogen	18.10
Sulphur Oxygen	24.37
	100.00

> This is the proteid commonly known as vegetable vitellin. It is precipitated from saline solutious by dilution and by dialysis, is not coagulated by heating below 90°, and above that temperature only partially. It is not precipitated by saturating its solution with sodium chlorid, but is thrown down from saline solutions by adding acid.
>
> IV. Hordein, a proteid insoluble in saline solutions, very slightly soluble in pure water, and extremely soluble in alcohol of about 75 per cent. This is barley proteid

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[a] J. Amer. Chem. Soc., 17: 447.
[b] J. Amer Chem. Soc., 17: 565.

described by Ritthausen as mucedin. It has almost the same physical and chemical properties as gliadin obtained from wheat and rye kernels but a different composition.

	Per cent.
Carbon	54.29
Hydrogen	6.80
Nitrogen	17.21
Sulphur	.83
Oxygen	20.87
	100.00

About 4 per cent of the seed consists of this substance.

V. After extracting the barley flour with salt solution and with alcohol the residue still contained 42 per cent of the total nitrogen, corresponding to proteid matter equal to about 4.5 per cent of the flour. It was not possible to extract more than a very small amount of this residual proteid with dilute potash water, as the treatment for removal of the other proteids rendered it soluble, if it were not so already.

VI. The barley flour contained 1.83 per cent of nitrogen, and if it be assumed that this all belonged to proteid matter with 17 per cent of nitrogen, the flour would contain 10.75 per cent of proteids. The barley accordingly contained about 4.5 per cent of insoluble proteid, 4 per cent of hordein soluble in dilute alcohol, 0.3 per cent of albumin, and 1.95 per cent of globulin and proteose.

THE PROTEIDS OF THE PEA, LENTIL, HORSE BEAN, AND VETCH.

The proteids of the leguminous seeds appear to be very much alike in their reactions and properties, though differing widely in the quantity of the various proteids found in the grains of this interesting group. Osborne and Campbell have summed up their properties as follows:[a]

LEGUMIN.

Legumin forms the chief proteid constituent obtainable from the vetch, pea, lentil, and horse bean. In the first-named seed about 10 per cent of the meal was found to consist of legumin; in the three other seeds this proteid is associated with vicilin, from which we have no method for its quantitative separation. From the pea about 10 per cent, from the lentil 13 per cent, and from the horse bean about 17 per cent of these mixed proteids were obtained. The lentil contains the least proportion of legumin, which seems to form about two-thirds of the mixed proteids, while the horse bean contains the greatest, as in this seed vicilin is present in relatively small amount.

Legumin is a globulin, for it dissolves readily in saline solutions, and is precipitated therefrom either by dialysis, dilution, or cooling. By dialysis or by cooling it separates in the form of spheroids which, after settling from the solution, unite to form a plastic mass. By diluting its concentrated solutions the legumin separates as a viscid, translucent fluid. This fluid, when treated with water, becomes opaque and solid, so that the legumin can be converted into a coarse meal by rubbing with a glass rod under water. Conglutin from lupin seeds and amandin from almonds behave similarly, as do gliadin of wheat and rye and hordein of barley, when precipitated from alcoholic solutions by dilution with water.

Solutions containing more than 2 per cent of sodium chlorid dissolve legumin abundantly; those containing less salt have a solvent power rapidly decreasing with the diminishing salt content, so that a 1 per cent salt solution dissolves very little. Saturation with sodium chlorid or magnesium sulphate does not precipitate legumin from its solution in brine, but saturation with sodium sulphate at 34° precipitates it almost completely.

In pure water legumin is entirely insoluble, but if the solution from which the legumin is precipitated contains acid, this may combine with the legumin, and the resulting preparation, like other acid globulins, will then dissolve in pure water.

If seeds containing legumin are extracted with water, more or less of the legumin is dissolved—from the pea about 4 per cent, from the vetch 2.5 per cent, from the lentil 10 per cent, and from the horse bean 16 per cent. The legumin thus dissolved is largely precipitated by dialysis in water, by the addition of acids and by lime salts and very slightly by great dilution with water. These aqueous extracts react strongly acid with litmus; and alkaline with lacmoid, a behavior doubtless due to acid potassium phosphates, together with organic acids or acid salts for acids combined with proteids do not react with lacmoid, although they readily turn blue litmus red. Solutions which we have made of legumin from various seeds, as well as of edestin from hemp seeds, by dissolving the proteid in monohydrogen potassium phosphate, have shown us all the reactions of these aqueous extracts excepting one presented by the horse bean, the unneutralized water extracts of which are precipitated with pure sodium chlorid. This reaction we can not explain; for these extracts give with dilute acids precipitates soluble in salt solutions and no precipitates on neutralization, reactions which seem to exclude the presence of acid globulin.

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[a] J. Amer. Chem. Soc., **20**: 410.

Solutions of legumin or of edestin in monohydrogen potassium phosphate behave more like solutions in dilute alkali carbonates than like solutions in neutral salts; for they give precipitates with dilute acids which are soluble in more acid or in salt solutions and are not precipitated by dilution unless very little phosphate is present. If phosphoric acid is added to the potassium phosphate solution, the solvent power of the phosphate is diminished and the facility with which the proteid is precipitated by dilution is increased; but if only little acid is added, yet enough to give a decided reaction with litmus, considerable quantities of the globulins are still dissolved and the reactions of solutions so made closely resemble those of the aqueous extracts of these seeds. As a result of numerous comparisons of the reactions of solutions so prepared with those of the aqueous extracts of leguminous seeds, we believe that the extraction of legumin by water from these seeds is due to the presence of acid potassium salts of phosphoric and organic acids, and that in consequence of the varying proportions of these substances in the different kinds of seeds, different amounts of legumin are thus extracted from them. As the proportion of phosphoric acid to potash in these leguminous seeds is much smaller than in most of the other seeds which we have examined, the character of the salts present may fairly be supposed to differ, and consequently the solubility of the proteids would also differ when the seeds are extracted with water. Thus in lupin seeds the amount of phosphoric acid, 1.4 per cent, exceeds that of the potash, 1.1 per cent, and from them water extracts but little proteid. Liebig and Ritthausen attributed the solubility of legumin to basic phosphates, and the latter undertook extensive experiments to show the presence of an excess of potash in the aqueous extracts; but such a supposition appears to overlook the fact that the aqueous extracts of these seeds are decidedly acid toward litmus.

Dissolved in dilute sodium chlorid solution, legumin is precipitated by a little acetic acid, the precipitate being soluble in an excess of sodium chlorid. The solubility of precipitates so produced depends on the relative proportions of salt and acid.

Legumin extracted without neutralizing the natural acid of the seed precipitated by dialysis, either directly or after precipitation with ammonium sulphate, is usually converted to a large extent into insoluble "albuminate."

This "albuminate" differs from the similar insoluble products obtained from most other globulins; for when treated with salt solution it becomes gelatinous, can not be filtered, and, on washing with water, shrinks, becomes opaque, and finally granular, so that it can be very readily washed on a filter. This substance appears to become hydrated by salt solution and dehydrated by pure water.

If the acid of the seed is previously neutralized, the globulin extracted by salt solution yields very little if any insoluble "albuminate," which indicates that the latter is a product of the action of the acid of the seed. This fact is in harmony with experiments described in a former paper of ours on the action of minute quantities of acid on globulins. In very dilute acids, and alkalies in absence of salts, legumin dissolves readily and abundantly, from which solutions, if at once neutralized, it is precipitated in a form soluble in sodium chlorid solution. By this treatment no evidence of change has been detected. Solutions made with hydrochloric acid are precipitated by a small excess of acid, but those made with acetic acid are not precipitated by any excess of acid.

Solutions of legumin in 10 per cent sodium-chlorid brine are not rendered turbid by long heating in a boiling water bath.

Dissolved in 10 per cent sodium-chlorid brine, legumin is precipitated by a very little hydrochloric acid, but a relatively considerable amount of acetic acid is required to produce a precipitate in such solutions.

Sodium-chlorid solutions of legumin give large precipitates with tannic acid, as well as with picric acid, those formed by the latter dissolving in an excess of salt solution if too much picric acid had not been previously added. With mercuric chlorid no precipitate is produced.

With nitric acid, Milton's and Adamkiewicz's tests, proteid reactions are obtained. With the biuret test a violet color is given, which on standing becomes a deep rose-red like that given by peptones.

Legumin has been supposed by some investigators to contain phosphorus, and therefore to belong with the nucleo-proteids. A careful testing of thoroughly purified samples, by fusing with caustic soda and nitrate, and treating the solution of the fusion acidified with nitric acid with ammonium molybdate, showed that in some of the preparations only just detectable traces of phosphorus were present, while other preparations contained none whatever.

Although we have examined large numbers of our preparations of the different plant proteids for phosphorus, we have as yet found none which in carefully purified samples contained more than a few hundredths of a per cent, a quantity so small that it is reasonable to consider it as a constituent of the ever-present ash.

The composition of legumin is shown by the following figures, which are averages of a number of preparations from each of the different seeds:

Legumin.

Constituents.	Pea.	Lentil.	Horse bean.	Vetch.	Average.
Carbon	51.74	51.73	51.72	51.69	51.72
Hydrogen	6.90	6.89	7.01	6.99	6.95
Nitrogen	18.04	18.06	18.06	18.02	18.04
Sulphur	.42	.42	.39	.43	.41
Oxygen	22.90	22.92	22.82	22.87	22.88
Total	100.00	100.00	100.00	100.00	100.00

VICILIN.

Vicilin is a globulin associated with legumin in the pea, lentil, and horse bean. But as we have no means of separating vicilin and legumin quantitatively, we can state nothing respecting the amount in which it occurs in these seeds further than that the lentil contains the most and the horse bean the least. In the lentil it probably forms about one-third of the mixed globulins. That vicilin is not derivative of legumin, is almost conclusively proved by the fact that no vicilin can be obtained from the vetch.

The most remarkable characteristic of vicilin is its content of sulphur, less than that of any other known proteid. This element, it may be noticed, diminishes in quantity with repeated precipitation, as though by this process sulphur were split off from the molecule. The total quantity, however, is so small that it might appear unsafe to draw conclusions from the differences observed; that is, from 0.23 per cent maximum to 0.08 per cent minimum. Yet, repeated determinations of this element have shown us that the differences were not analytical. It would seem possible by sufficiently repeated precipitation to obtain from this preparations free from sulphur.

In salt solution vicilin is much more soluble than legumin, so that by repeated precipitation from diluted solutions the two globulins can be separated.

When solutions of vicilin in 10 per cent brine are heated in a water bath they become turbid at 90°, and at 95° flocks separate. When heated for some time at 100° this globulin is almost completely coagulated. In water vicilin is insoluble. In 1 per cent sodium chlorid solution it dissolves considerably, while in slightly stronger solutions it is much more soluble, the solution of the globulin appearing to depend on the presence of enough salt to form a soluble compound.

In its reactions it very closely resembles legumin.

The composition of vicilin we found to be as follows:

Vicilin.

Constituents.	Pea.	Lentil.	Horse bean.	Average.
Carbon	52.36	52.13	52.38	52.29
Hydrogen	7.03	7.02	7.04	7.03
Nitrogen	17.40	17.38	17.52	17.43
Sulphur	.18	.17	.15	.17
Oxygen	23.03	23.30	22.91	23.06
Total	100.00	100.00	100.00	100.00

LEGUMELIN.

We have found legumelin in all leguminous seeds which we have examined with the exception of the white bean (*Phaseolus vulgaris*) and the blue and yellow lupin.

It is difficult to decide whether this proteid should be considered an albumin or a globulin.

By prolonged dialysis of solutions containing considerable legumelin a very small part separates, usually in a form insoluble in salt solutions. In one case we obtained a precipitate by dialysis which was wholly soluble in brine, to a solution from which a relatively large coagulum separated on heating to 65°, thus indicating a globulin. We have found, however, that other proteids, notably edestin, undergo a change in solubility, whereby the proteid becomes gradually more and more difficult to dissolve. Thus edestin changes from a form insoluble in cold brine to one soluble in hot brine, also to one soluble in hot brine to one insoluble therein, but soluble in dilute sodium carbonate solution, and finally from a form insoluble in alkali carbonate to one soluble only in caustic alkali. It is not unreasonable to expect that a proteid which might properly be considered an albumin (i. e., soluble in pure water and coagulable on heating) should undergo a similar change whereby it becomes no longer soluble in pure water but dissolves in salt solutions and then further changes to a form no longer soluble in brine but dissolved by alkali carbonates.

Such a very large proportion of the legumelin resists precipitation by dialysis, that we are inclined to regard the small precipitates which we have obtained in this way as due to changes similar to those which produce so-called "albuminates" from globulins.

In a paper on the proteids of the cowpea[a] we designated this proteid as a globulin, but in view of our subsequent, much more extensive experience we now consider legumelin to be more properly classed with the albumins.

The amount of legumelin which we have found in the seeds discussed in this paper was in the pea 2 per cent, vetch, 1.5 per cent; lentil and horse bean, 1.25 per cent.

No definite coagulation point can be stated for legumelin, as the presence of salts or acids as well as the proportion of dissolved legumelin have a great effect on the temperature at which coagulation takes place. * * *

In the following table we give the average of analyses which we have made of coagulated legumelin from different seeds. These preparations have been obtained in a variety of ways, as can be seen by consulting our papers on the seeds named.

Legumelin.

Constituents.	Pea.	Lentil.	Horse bean.	Vetch.	Adzuki bean.	Cow-pea.	Soy bean.	Average.
Carbon	53.31	53.22	53.03	53.31	53.97	53.25	53.06	53.31
Hydrogen	6.99	6.82	6.97	6.97	7.01	7.07	6.94	6.97
Nitrogen	16.30	16.27	16.22	16.24	16.31	16.36	16.14	16.26
Sulphur	1.06	.94	1.30	1.11	.88	1.11	1.17	1.08
Oxygen	22.34	22.75	22.48	22.37	21.83	22.21	22.69	22.38
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

PROTEOSE.

As the proteose of these seeds is present in small amount and is difficult to obtain pure, we have not as much information respecting it as is desirable. The pea appears to contain about 1 per cent, the horse bean about 0.5 per cent, and the lentil and vetch evidently less. It is probable that more or less of this proteose may be lost by diffusion, for 10.5 grams of what was doubtless nearly pure proteose from the pea after solution and dialysis yielded only about 6 grams when precipitated.

We have obtained a few of the reactions of proteose from the pea and horse bean, but no reactions of this proteid from the lentil and vetch.

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[a] J. Amer. Chem. Soc., 19: 499.

Proteose.

Constituents.	Pea.		Lentil, No. 68.	Horse bean.		Vetch, No. 22.
	No. 46.	No. 47.		No. 89.	No. 98.	
Carbon	50.24	49.66	50.17	50.24	49.96	50.85
Hydrogen	6.76	6.78	6.77	6.66	6.76	6.75
Nitrogen	17.35	16.57	16.81	17.11	16.95	16.65
Sulphur	1.25	1.40	1.27	1.87	2.75	25.75
Oxygen	24.40	25.59	23.98	24.12	23.58	....
Total	100.00	100.00	100.00	100.00	100.00	100.00

If the difficulty encountered in purifying these preparations of proteose and the different methods by which they have been obtained are considered, the agreement between them, except for the sulphur in those from the horse bean, makes it probable that these figures nearly represent the composition of this substance.

PROTEIDS OF THE MAIZE KERNEL.

The proteids of corn have been investigated by Osborne and Chittenden.[a] These proteids may be distinguished according to their solubilities as follows:

(*a*) Proteid, soluble in pure water, having some of the properties of proteose.

(*b*) Globulins, insoluble in pure water, but soluble in salt solutions. Edestin and maysin.

(*c*) Proteid, insoluble in water or salt solutions, but soluble in alcohol of 60 to 99 per cent. Zein.

(*d*) Proteid matter, insoluble in water, salt solution, and alcohol, but soluble in dilute alkalies and acids.

Later Osborne[b] determined the quantities of these proteids in corn. One hundred grams of the yellow corn meal contained, approximately, as follows:

		Grams.
Proteid soluble in 0.2 per cent potash	3.15 grams containing 15.82 per cent N.	=0.4983
Zein	5.00 grams containing 16.13 per cent N.	=0.8065
Very soluble globulin	0.04 grams containing 15.25 per cent N.	=0.0061
Edestin	0.10 grams containing 18.10 per cent N.	=0.0181
Maysin	0.25 grams containing 16.70 per cent N.	=0.0417
Proteose	0.06 grams containing 17.00 per cent N.	=0.0102
		1.3809
Nitrogen undissolved by dilute potash water		0.1645
Total		1.5454
Nitrogen in meal by analysis		1.5400
Mean percentage of nitrogen in maize proteids		16.057

The various proteids that have been separated from some other vegetable substances might be stated, but enough has been given to indicate the general characteristics of the proteids of vegetable substances. Reference has been made to the readiness with which these bodies change when in a moist condition or when they are in solution, and to the fact that they combine with acids, forming new substances with new properties. In addition to this, the solubilities of the separate proteids will not permit of the extraction of one proteid without being more or less contaminated with another. For general comparative purposes, however, the separation of the proteids into water-soluble, salt-soluble, alcohol-soluble, and insoluble proteids gives valuable results. The following method proposed by Prof. H. H. Snyder[a] for the separation of the proteids of flour, based largely on the methods employed by Osborne and Voorhees will apply fairly well to all vegetable substances.

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[a] Amer. Chem. J., 18: 453, 529.

[b] J. Amer. Chem. Soc., 19: 532.

Water-soluble nitrogen.—Fifteen grams of flour were weighed into a flask and 250 cc distilled water added, the temperature being kept at about 30° by placing the flask in water of that temperature. The flask was shaken occasionally. After two hours, 200 cc of the clear filtrate were placed in a Kjeldahl digestion flask containing a little sulphuric acid and evaporated nearly to dryness, when the remainder of the acid necessary for oxidation was added and the determination of nitrogen completed in the usual way. When it was also desired to separate water-soluble gliadin 30 grams of flour and 500 cc of water were used. One-half of the filtrate was evaporated nearly to dryness and the residue treated with 70 per cent alcohol to extract the soluble gliadin. The extract was filtered through a small filter. The filter paper with the insoluble proteid residue was placed in a Kjeldahl flask and the nitrogen in it determined in the usual way.

Salt-soluble nitrogen.—After removing the water-soluble proteids the flour residue was placed in a flask and 500 cc of 5 per cent sodium chlorid solution added. After standing for about two hours at 30° the nitrogen in the extract was determined as in the case of proteid soluble in water.

Gliadin.—Five grams of flour or other material were weighed into a flask and 250 cc of 70 per cent alcohol added, the contents of the flask being shaken at intervals of half an hour for three hours. The alcohol was left in contact with the flour for about eighteen hours. The alcoholic extract was then filtered and 100 cc of the filtrate placed in a Kjeldahl digestion flask connected with a condenser. The alcohol was removed by distilling and the nitrogen in the residue determined in the usual way. With old or unsound flours a correction must be made for soluble amid bodies.

Glutenin.—The residue from the gliadin determination was washed with 70 per cent alcohol until the washings no longer gave a reaction for proteids. It was then transferred to a flask and 200 cc of a 5 per cent solution of sodium chlorid added to remove globulin and other proteids. After two hours' extraction the residue was washed with distilled water and transferred to a flask, 250 cc of a 0.2 per cent solution of potassium hydroxid added, and after three hours' extraction the solution filtered, and the nitrogen in 200 cc of the filtrate determined in the usual way. The glutenin determination is the most troublesome of all, because the other proteids must be removed before it can be extracted with potassium hydroxid. For all practical purposes the glutenin may be estimated by difference. Results thus obtained are within 0.1 per cent of the actual amount recovered with potassium hydroxid.

Amid nitrogen.—Fifteen grams of flour were treated as described for the determination of water-soluble proteids. Fifteen cubic centimeters of Stutzer's copper solution were added to 200 cc of the filtrate and the albuminoid nitrogen determination was made in the usual way. The difference between the nitrogen of the water-soluble proteid and the albuminoid nitrogen represents the amid nitrogen.

Tannic acid was also used to precipitate the total soluble proteid matter, and gave satisfactory results. The usual method for the determination of albuminoid nitrogen is not applicable to flour because a paste-like mass is formed which can not be readily filtered.

In following out this method in analyses made in our own laboratory we had no difficulty in filtering off the water extract from leguminous grains, but we found it practically impossible with flour. However, I think these separations could be made by means of centrifugal force. We have not been able to make a thorough test of this method, so have not thought it well to give any of the results obtained.

The PRESIDENT. The report of the associate referee on meat proteids is now in order.

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[a] Minn. Exp. Sta. Bul. No. 63, 1899.

REPORT ON SEPARATION OF MEAT PROTEIDS.

By W. D. Bigelow, *Associate referee.*

This subject has been again divided into the nitrogenous constituents of meats, and the nitrogenous constituents of meat extracts. The associate referee has been fortunate in securing the collaboration of Mr. Grindley, who has devoted so much attention to the study of meats and includes in his work a careful comparison of the amount of proteids precipitated by various reagents in the water extracts of meats, both raw and cooked. Mr. Grindley's report is submitted as a separate paper.

During the last year the study of the separation of the proteids in meat extracts, which has been conducted in the food laboratory of the Bureau of Chemistry, was continued by W. D. Bigelow and F. C. Cook for the purpose of confirming previous results, and of studying the action of combinations of reagents which had not previously been tried in that laboratory. Before reporting on the work of the past year it has been thought best to give here, in greater detail than has already been published, the results of previous work by the referee and R. Harcourt.

Considerable attention has been given in recent years to the method for the precipitation of proteids by means of bromin, as suggested by Allen and Searle,[a] and modifications of that method which have since been published. A careful study was made of this method with a view to determining the conditions under which it would yield the maximum results. After repeated trials it was decided that the filtration could not be successfully performed under less than two hours after precipitation. We were unable to filter the mixture under one-half hour, as directed by Allen and Searle, owing to the incomplete separation of the precipitate. Aliquot parts of the filtrates from the proteids coagulated by heating were subjected to the action of bromin water in some cases, and in others saturated with bromin. In all cases the solutions were acidified with hydrochloric acid, as directed by Allen and Searle, before the addition of the bromin water or bromin.

Table I.—*Bromin precipitate in filtrate from coagulated proteids.*

Description of sample.	Precipitate by bromin water filtered after 2 hours.	Precipitate by saturation with bromin filtered after 16–18 hours.
	Per cent.	*Per cent.*
Hammond's beef extract	1.32	1.31
A. & J.'s liquid peptones	.04	.04
Tropon	.05	.06
Arlington Chemical Co.'s beef peptones	.22	.34
Arlington Chemical Co.'s liquid peptonoids	.03	.09
The London Essence Co.'s essence of chicken	1.32	1.26
Rose's peptonized beef	2.40	2.52
Extractum carnis Liebig	.99	.86

It would appear from the figures given above that the results by allowing the liquid to stand over night before filtration are practically identical with those obtained by filtering within two hours after precipitation. The former method gives filtrates that are more uniformly clear, and it can be used with greater economy of time. It was therefore chosen for subsequent work in comparison with other methods. The details employed were those published in Bulletin 65 of the Bureau of Chemistry, page 11.

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[a] Analyst, 1897, 22: 259.

Phosphotungstic acid was suggested by Stutzer. It has long been conceded that all proteids except peptones were precipitated by ammonium sulphate.[a] Bömer[b] substitutes zinc sulphate for ammonium sulphate. He acidifies the filtrate from the insoluble and coagulable material with 1 cc of 1—4 sulphuric acid to prevent the precipitation of zinc phosphate, and saturates the solution with zinc sulphate. The precipitate is washed with a saturated solution of zinc sulphate and its nitrogen content determined by the Kjeldahl method. It is our purpose here to record the results of a careful study of the foregoing methods.

For the purpose of comparison Mallet's method was employed in the work reported herewith. In some cases a preliminary addition of tannin was made, and in others the tannin was omitted and the phosphotungstic acid added directly to the filtrate from the coagulated proteids. It is regretted that Schjerning's method, published while the work here reported was in progress, in which tannin and sodium chlorid are used as precipitants, did not come to our attention in time to be included.

The action of flesh bases of the reagents employed in the various methods was also studied. Samples of glycocin, leucin, alanin, asparogin, glutamin, tyrosin, betain, kreatin, kreatinin, hypoxanthin (sarkin), carnin, and allantoin, were obtained in as high a state of purity as practicable, dissolved in water acidified with hydrochloric acid and successive small portions of the solution treated with bromin, tannin, phosphotungstic acid, ammonium sulphate, and zinc sulphate. No precipitation occurred in any case with bromin, ammonium sulphate, or zinc sulphate. Tannin gave a very slight turbidity with alanin and betain.

On boiling the solution and allowing it to cool, this insoluble matter subsided and was separated by filtration. Further concentration of the filtrate did not cause a reappearance of this turbidity, which was evidently due to a slight trace of an impurity which had not been removed from the preparations employed.

Phosphotungstic acid either produced no precipitate or a precipitate which dissolved on heating. When the solutions were heated to from 85° to 90° no insoluble matter was left in any case, except that alanin and betain retained a scarcely perceptible turbidity, as they did with tannin. As was stated, under tannin this was doubtless due to a trace of an impurity in the preparations employed. These tests are in part a repetition of Mallet's work, but it is deemed appropriate to mention them here in confirmation of his results.

The reagents mentioned were then employed for the examination of a number of samples of meat extracts and similar products, the results of which are shown in Table II. The percentage of material insoluble in water, of material coagulated by boiling after slightly acidifying, were first determined by the methods outlined in Bulletin 65 of the Bureau of Chemistry. Aliquot parts of the filtrate from the proteids coagulated by boiling were then treated with the reagents mentioned above. It will be observed that no relation exists between the proteids precipitated by bromin and those precipitated by zinc sulphate.

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[a] Stutzer, Zeit. anal. Chem., 1895, 5: 372.

[b] Zeit. anal. Chem., 1895, 5: 562; Bureau of Chemistry Bul. No. 65, p. 18.

TABLE II.—*Nitrogenous bodies in meat extracts* (*Bigelow and Harcourt*).

[Expressed in terms of total nitrogen in original sample.]

Serial No.	Substance.	Total.	Insoluble in water.	Precipitate by boiling in slightly acid solution.	Precipitate (from filtrate by boiling) by—					
					Bromin.	Zinc sulphate.	Bromin filtrate in zinc sulphate.	Zinc sulphate + bromin.	Phosphotungstic acid.	Bromin and phosphotungstic acid.
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
18239	Beef extract, paste	8.43	....	....	13.88	6.168	....	....	63.82	58.36
18631	Liquid food	1.90	14.74	79.48	.00	.00	.00	.00	1.579	1.579
18633	Meat powder, digested	12.13	.495	....	83.51	82.00	4.1	86.1	91.92	66.78
19398	Beef extract, paste	8.88	2.252	.34	14.75	12.95	3.378	16.44	....	23.65
19399	Beef extract, fluid	3.04	....	....	3.29	1.65	8.22	9.86	10.53	22.36
19456	Meat powder, digested	14.41	94.59	.14	.42	.42	....	....	....	.56
19457	Liquid peptone	.25	.00	8.00	12.00	8.00	8.00	16.00	....	24.00
19458	Beef peptonoids, powder	4.27	68.62	10.77	7.96	8.43	2.81	11.24	....	10.54
19459	Liquid peptonoids	.34	.00	.00	26.47	8.82	17.65	26.47	....	32.55
19460	Meat powder	12.03	57.19	.17	15.71	8.90	....	....	23.69	22.69
19461	Commercial peptone, fluid	.95	.00	.00	34.74	18.95	....	....	52.63	44.21
19462	Beef tonic, fluid	.19	.00	.00	21.05	68.42	....	....	42.10	42.10
19463	Beef extract, powder	2.05	50.24	7.81	8.29	40.00	....	....	15.61	14.63
19464	Essence of chicken, fluid	3.43	....	2.04	36.71	27.39	33.51	60.90	69.64	....
19465	Fluid beef, paste	7.15	11.05	.00	29.79	40.98	11.75	52.78	59.86	62.10
19466	Beef extract, paste	3.23	50.16	.00	21.05	21.36	....	....	17.64	28.48
19467	Peptonized beef, gelatinous	6.27	11.48	2.39	27.91	36.52	....	....	74.32	85.96
19766	Beef juice pressed at 60°.	1.09	20.18	24.77	11.01	38.53	....	....	14.68	21.10
19767	.....do	1.18	11.02	33.90	10.17	16.93	....	....	8.47	15.25
19785	Beef juice pressed cold	.48	....	70.83	4.17	6.25	....	....	6.25	8.33
19786	.....do	.43	....	69.76	4.65	2.33	....	....	6.98	6.98
19787	Beef juice pressed after heating 6 hours	.24	....	....	25.00	20.83	....	....	50.00	41.67
19817	Liquid peptone	.31	.00	8.68	9.68	.00	....	....	32.26	22.58

Considering the statements of Allen and Searle, that all proteids, including peptones, were precipitated by bromin, it was intended to precipitate albuminoses by zinc sulphate, and albuminoses and peptones combined by bromin, and to calculate the percentage of peptones by the difference between the amount of albuminoses and peptones present. It was soon found, however, that not only was this difference not equal to the percentage of peptones present, but that frequently the zinc sulphate precipitate was greater in amount than the bromin precipitate. The filtrate from the zinc sulphate precipitate in the case of samples whose examination had not been completed was diluted with an equal volume of water, as directed by Allen, saturated with bromin, and allowed to stand over night, after which the precipitated nitrogenous compounds were separated by filtration, and the amount of nitrogen present determined. It was found that the nitrogen precipitated by bromin in the filtrate from the zinc sulphate precipitate was sometimes greater than was precipitated from the original solution. The sum of the nitrogen precipitated by zinc sulphate and precipitated by bromin in the filtrate from the same is usually less than that precipitated by phosphotungstic acid, although in many cases it is not materially less. It is probable that in products containing a small amount of peptones the results so obtained are sufficiently near the truth. On the other hand, in the case of commercial peptones and of meat products which have been heated for a long period, or peptonized in any manner, the use of bromin does not give reliable results even in the filtrate from the zinc-sulphate precipitate.

It is worthy of remark that, while Allen and Searle stated that all proteids were completely precipitated by bromin they only reported results obtained in the filtrate from the zinc-sulphate precipitate, and, as stated above, such results when added to the zinc sulphate precipitate are much higher than those obtained by bromin in the original solution. At the same time the nitrogen precipitated by bromin is always too low.

Since the partial publication of this work these observations on the unreliability of bromin for determining nitrogen have been confirmed in different classes of proteids by Fraps and Bizzell[a] and by Van Slyke,[b] while Schjerning[c] had previously noted that fact.

The precipitate by phosphotungstic acid was usually higher than that obtained by zinc sulphate, although in some cases it was considerably lower. The combined precipitates obtained with tannin and phosphotungstic acid did not have the effect of precipitating the peptones which were not precipitated by phosphotungstic acid, but on the other hand, gave in some cases a lower result than with the reagent alone. As stated before, this work was done before the publication of Schjerning had come to our attention, and salt was not added to the tannin reagent.

The results of this work as a whole were unsatisfactory. There appears to be no agreement between the results of different methods, and it does not seem that the products in themselves are sufficiently different in their nature to warrant the discrepancy in the results obtained. In order to obtain additional data regarding the matter, and for the purpose of comparing the tannin-salt reagent with the methods already tried, and also to study the precipitates obtained by various combinations of the reagents in use, additional work was undertaken during the last summer by W. D. Bigelow and F. C. Cook. It is felt that so far only a beginning in this work has been made.

Eight commercial products were taken of which 4 were ordinary meat products of pasty consistency; one a so-called beef juice; one a well-known digested meat powder, and two commercial peptones. A solution of each product was made, and aliquot portions were subjected to the action of bromin, zinc sulphate in saturated solution, phosphotungstic acid above 90°, and tannin-salt, respectively. The filtrate from the phosphotungstic acid reagent was allowed to cool and again filtered, and the amount of nitrogen obtained in the filtrate was added to that precipitated by the phosphotungstic acid in hot solutions, the same being given as phosphotungstic acid in the cold. The results obtained are given in Tables III and IV.

TABLE III.—*Nitrogenous bodies in meat extracts* (*Bigelow and Cook*).

[Expressed in terms of total nitrogen in original sample].

PRECIPITATES FROM ZINC SULPHATE FOLLOWED BY OTHER REAGENTS.

	Description of sample.	Zinc sulphate.	Zinc sulphate +bromin.	Zinc sulphate + phosphotungstic acid.	Zinc sulphate, +tannin-salt.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1	Meat extract	21.1	25.5	31.4	21.6
2	....do	20.4	27.6	40.3	21.0
3	....do	10.8	22.8	....	11.7
4	....do	46.1	52.6	62.5	46.5
5	Meat powder, digested	81.1	85.2	88.7	....
6	Commercial peptone	19.9	42.2	58.9	....
7	....do	42.0	44.1	76.4	....
8	Beef juice	6.2	9.9	14.3	....

[a] J. Amer. Chem. Soc. 1900, **22**: 709.
[b] U. S. Dept. of Agri., Bur. of Chem. Bul. No. 73, p. 96.
[c] Zeit. anal. Chem., 1900, **39**: 545.

TABLE III.—*Nitrogenous bodies in meat extracts*—Continued.

PRECIPITATES FROM PHOSPHOTUNGSTIC ACID FOLLOWED BY OTHER REAGENTS.

Number of sample.	Phosphotungstic acid.	Phosphotungstic acid +bromin.	Phosphotungstic acid +zinc sulphate.	Phosphotungstic acid +zinc sulphate. +bromin.	Phosphotungstic acid +tannin-salt.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	41.5	44.0	42.2	44.1	41.5
2	41.7	43.3	42.5	44.6	41.7
3	40.3	42.8	41.2	43.6	40.3
4	70.4	71.4	70.8	71.5	70.4
5	93.5	93.9	98.6	104.3	95.2
6	....	....	....	....	....
7	56.0	56.6	67.9	85.0	61.1
8	26.0	26.2	28.8	31.2	28.1

PRECIPITATES FROM TANNIN-SALT FOLLOWED BY OTHER REAGENTS.

Number of sample.	Tannin-salt.	Tannin-salt+bromin.	Tannin-salt +zinc sulphate.	Tannin-salt +zinc sulphate +bromin.	Tannin-salt +phosphotungstic acid.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	32.6	39.0	58.5	61.3	35.8
2	39.9	43.4	53.6	56.2	41.7
3	35.4	41.1	52.4	55.8	37.3
4	66.9	....	76.0	77.5	....
5	89.4	97.0	100.5	101.5	....
6	53.9	62.1	76.2	77.8	....
7	78.4	84.6	93.1	94.0	....
8	17.0	26.3	33.1	36.3	....

TABLE IV.—*Rearrangement of figures in Table III for purposes of comparison.*

[Nitrogenous bodies in meat extracts expressed in terms of total nitrogen in original sample.]

PRECIPITATES FROM THE FOUR REAGENTS SEPARATELY.

	Description of samples.	Zinc sulphate.	Phosphotungstic acid.		Tannin-salt.	Bromin.
			Hot.	Cold.		
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	Meat extract	21.1	41.5	42.48	32.6	23.9
2	.....do	20.4	41.7	42.22	39.9	26.2
3	.....do	10.8	40.3	40.78	35.4	14.1
4	.....do	46.1	70.4	70.63	66.9	40.2
5	Meat powder, digested	81.1	93.5	98.42	89.4	53.1
6	Commercial peptone	19.9	....	....	53.9	18.1
7	.....do	42.0	56.0	59.20	78.4	29.9
8	Beef juice	6.2	26.0	29.72	17.0	4.6

TABLE IV.—*Rearrangement of figures in Table III, etc.*—Continued.

PRECIPITATES FROM COMBINED REAGENTS, WITH COMPARISON OF THE ORDER IN WHICH USED.

Number of sample.	Zinc sulphate+phosphotungstic acid.	Phosphotungstic acid +zinc sulphate.	Tannin-salt+phosphotungstic acid.	Phosphotungstic acid+tannin-salt.	Zinc sulphate+tannin-salt.	Tannin-salt+zinc sulphate.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	31.4	42.2	35.8	41.5	21.6	58.5
2	40.3	42.5	41.7	41.7	21.0	53.6
3	....	41.2	37.3	40.8	11.7	52.4
4	62.5	70.8	....	70.4	46.5	76.0
5	88.7	98.6	....	95.2	....	100.5
6	58.9	....	....	....	....	76.2
7	76.4	67.9	....	61.1	....	93.1
8	14.3	28.8	....	28.1	....	33.1

PRECIPITATES FROM THE SEVERAL REAGENTS FOLLOWED (1) BY BROMIN, (2) BY ZINC SULPHATE AND BROMIN.

Number of sample.	Zinc sulphate+bromin.	Phosphotungstic acid+bromin.	Tannin-salt+bromin.	Phosphotungstic acid+zinc sulphate +bromin.	Tannin-salt+zinc sulphate +bromin.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	25.5	44.0	39.0	44.1	61.3
2	27.6	43.3	43.4	44.6	56.2
3	22.8	42.8	41.1	43.6	55.8
4	52.6	71.4	....	71.5	77.5
5	85.2	93.9	97.0	104.3	101.5
6	42.2	....	62.1	....	77.8
7	44.1	56.6	84.6	85.0	94.0
8	9.9	26.2	26.3	31.2	36.3

The filtrates from the precipitates obtained by zinc sulphate, phosphotungstic acid, and tannin-salt were then divided into aliquot parts, and one part from each was treated with all four of the reagents for the purpose of determining what combination would give the highest result. It was the intention also to treat these various combinations of reagents with the ordinary simpler amids, as was done in connection with the previous work. Up to the present time, however, this has not been accomplished.

In this work the amount of nitrogen precipitated by phosphotungstic acid, even in hot solutions, was greater than that precipitated by any other single reagent, except in the case of products known to contain a relatively large amount of peptones. The precipitate obtained with phosphotungstic acid in the cold was not appreciably greater than that obtained in hot solutions. Our previous results and those of others regarding the unreliability of bromin alone are confirmed. The combined zinc sulphate and bromin precipitate are, as formerly, considerably greater than either precipitate alone. On the other hand, the sum of the phosphotungstic acid and bromin precipitates is greater in all cases than the sum of the zinc sulphate and bromin precipitates. The highest results from the combination of any two reagents were obtained by tannin-salt followed by zinc sulphate in the filtrate from the same. By adding bromin to the diluted filtrate from the zinc-sulphate precipitate from the tannin-salt precipitate an additional precipitate is obtained, which, however, is so slight in almost all cases that it may be disregarded. The order in which the various reagents are employed has a material influence on the total amount of nitrogen precipitated by both; for instance, considerably more nitrogen is precipitated by phosphotungstic acid followed by zinc sulphate than by zinc sulphate followed by phosphotungstic acid. Again, while the highest results were obtained by the tannin-salt reagent followed by zinc sulphate, the lowest of all the combinations employed resulted from the use of zinc sulphate followed by the tannin-salt reagent.

These results are submitted merely as preliminary. They have not been confirmed, and have been obtained on so few samples that the reporter feels that no conclusion should be drawn from them. The discrepancies in the results of two reagents added in different order are surprising, and should be confirmed before being accepted. At the same time the general agreement among them suggests the advisability of continued study of the combinations of the various reagents employed.

At 12.40 the association adjourned until 1.30 p. m.

FRIDAY—AFTERNOON SESSION.

The President. The discussion of the subject of meat proteids will be continued by receiving Mr. Grindley's paper.

A STUDY OF THE NITROGENOUS CONSTITUENTS OF MEATS.

[A report to W. D. Bigelow, referee on food adulteration.]

By H. S. Grindley.

In the following studies the fresh substance of raw and cooked meats were used entirely. In other words, the raw and cooked meats were not air-dried for analysis. In order that the character and nature of the work here reported may be more easily and thoroughly understood, the methods which were used are given in detail. It should be clearly understood that the work here reported was undertaken entirely for the purpose of research and not for commercial analysis, and therefore the prime object has been to obtain definite information as to the value and accuracy of the several methods studied, regardless of the amount of time or labor required in doing it.

Preparation and Analysis of Cold-Water Extracts of Meats.

(a) *Preparation of cold-water extracts.*

Weigh off 9 portions of about 12.5 to 15 grams each. Transfer to beakers holding about 400 cc and add 100 cc of ammonia-free water to each lot. Stir mixture thoroughly for 30 minutes. Allow to settle for a few minutes (5–10) and filter through an 11 cm ashless and nitrogen-free filter. Collect the filtrates in 500 cc measuring flasks. See that the funnels touch the sides of the flasks so that the filtrates do not drop directly upon the liquid but flow slowly down the sides of the flasks.

Treat residues with successive portions of ammonia-free water, using about 50 cc for each extraction, stirring for 15 minutes during each extraction, and after each extraction allow the solid particles to settle for from 5 to 10 minutes before filtering. Continue this treatment until each filtrate measures about 470 cc.

Be sure each time to drain off thoroughly all the water from the residue in the beaker and also be sure to let the water upon the filter paper used in each extraction run entirely through before adding the water from another extraction. Also be careful to wash the filter paper and the residue upon the same thoroughly with each extract. These precautions are necessary in order to insure complete extraction.

Transfer carefully the extracts of about 470 cc each to a clean, dry 2-liter measuring flask, washing out each 500 cc flask twice, using about 10 cc of distilled water each time. Transfer the measuring extracts to a 5-liter flask and mix thoroughly without unnecessary agitation. Immediately measure out the volumes given below for the following determinations:

(b and c) Total solids and ash: Take 3 portions of 100 cc each.

(d) Total nitrogen: Take 3 portions of 100 cc each.

(e, f, l) Nitrogen in the form of coagulable proteids, albumoses by zinc sulphate and peptones by bromin in the filtrate from the precipitate produced by zinc sulphate: Take 3 portions of 200 cc each.

(g) Nitrogen precipitated by tannin and sodium chlorid: Take 3 portions of 200 cc each.

(h) Nitrogen precipitated by phosphotungstic acid in the cold: Take 3 portions of 200 cc each.

(i) Nitrogen in the form of ammonia: Take 3 portions of 200 cc each.

(j) Nitrogen precipitated by phosphotungstic acid in the hot: Take 3 portions of 200 cc each.

(k) Nitrogen precipitated by bromin in the cold-water extract: Take 3 portions of 200 cc each.

(l) See above (e).

(b) *Determination of total solids.*

Take 100 cc portions in triplicate. Evaporate to dryness in weighed platinum dishes upon the water bath. Clean sides and bottoms of dishes thoroughly. Heat in water oven for periods of exactly one hour until the weight is approximately constant.

(c) *Determination of ash.*

Ignite the dry residue obtained in (b) carefully over the free flame at a very low red heat until colorless or nearly so. Weigh quickly and heat again at a low red heat for only a few minutes (2 or 3) and then weigh again. Repeat this last treatment until weight is constant.

(d) *Determination of total nitrogen.*

Take 100 cc portions in triplicate and determine nitrogen very carefully as usual by the Kjeldahl method.

(e) *Nitrogen precipitated in the form of coagulated proteids in neutral solution.*

Take 200 cc portions in triplicate. Make the three portions exactly neutral to litmus paper by the addition of a N/10 solution of sodium hydrate or a N/10 solution of acetic acid. Heat the neutral solutions upon a vigorously boiling water bath stirring frequently so as not to allow the solution to foam over or to carry up the coagulated proteid upon the sides of the beaker. Continue the heating until any coagulum that forms settles completely, leaving a clear supernatant liquid. (The evaporation was usually continued until the volume of the original solution was reduced about one-half.) Filter the hot solution immediately through a well-fitting good nitrogen-free filter paper. Wash the beaker, filter, and precipitate thoroughly with boiling hot water. Determine nitrogen in the residue by the Kjeldahl method, being very careful to get off all the coagulated proteid from the beaker in which the precipitation was brought about. Evaporate the filtrate and washings to a volume of 30 cc upon the water bath and note whether or not further coagulation occurs. If coagulation occurs again filter, wash, and test as above. Save the filtrate for the following determination.

(f) *Determination of nitrogen precipitated by zinc sulphate as albumoses.*

Take the filtrates obtained from the above determinations in (e) and evaporate to a volume of 30 cc, cool to room temperature, add 1 cc of 50 per cent sulphuric acid, completely saturate with crystallized zinc sulphate. Be certain the solutions are completely saturated. About 17 grams of powdered zinc sulphate are necessary for the saturation of each 10 cc of liquid present. The saturated liquid must contain a slight excess of zinc sulphate, but much of an excess must be avoided, as it is likely to cause bumping in the subsequent determination of nitrogen. After excess of zinc sulphate has been added in the cold, warm the solution on the water bath until the solution is perfectly clear. Stir frequently while warming. Cool the mixture completely and filter through a 9-cm nitrogen-free filter paper. Wash the beaker, precipitate and filter paper thoroughly with a completely saturated solution of zinc sulphate, which is very slightly acidified with sulphuric acid (5 cc of concentrated sulphuric acid for 1,000 cc). Determine the nitrogen in the zinc sulphate precipitate by the Kjeldahl method. Save the filtrate and determine the nitrogen precipitated in the same by bromin as directed below in (l).

(g) *Determination of nitrogen precipitated by tannin and sodium chlorid.*

Take 200 cc portions in triplicate. Transfer the same to 250 cc measuring flasks, add to each 1 gram of pure sodium chlorid and a solution containing 12 per cent of tannin, until 1 drop added to the clear supernatant liquid gives no further precipitate. Dilute to the 250 cc mark, shake thoroughly, allow to stand 24 hours, filter through a dry filter, and determine the amount of nitrogen in 200 cc of each filtrate. This gives the amount of nitrogen in the form of creatin, amido-acids, and ammonium compounds not precipitated by tannin and salt. The amount of nitrogen precipitated by tannin and salt is obtained by subtracting from the total nitrogen in the aqueous extract the amount of nitrogen found in the filtrate from the tannin and salt.

The amount of nitrogen in the form of peptones is determined by difference; that is, by subtracting from the total nitrogen in the cold water solution the combined amounts of nitrogen found in (e), (f), and (g).

(h) *Determination of nitrogen precipitated by phosphotungstic acid in the cold.*

Take 200 cc portions in triplicate. Transfer the same to 250 cc measuring flasks, add to each 5 cc of 50 per cent of sulphuric acid, and shake thoroughly. To this add phosphotungstic acid (prepared as directed by Wiley) until 1 drop gives no further precipitation in the clear supernatant liquid. Dilute to the 250 cc mark and filter through a dry filter. Determine the amount of nitrogen in 200 cc of each filtrate. This gives the amount of nitrogen in the form of creatin, amido-acids, and ammonium compounds not precipitated by phosphotungstic acid. The amount of nitrogen precipitated by phosphotungstic acid is obtained by subtracting from the total nitrogen in the aqueous extract the amount of nitrogen found in the filtrate from the phosphotungstic precipitate. The amount of nitrogen in the form of peptones is determined by difference; that is, by subtracting from the total nitrogen in the cold water solution the combined sum of the amounts of nitrogen found in (e), (f), and (h).

(i) *Determination of nitrogen as ammonia.*

Take 200 cc portions in triplicate. Transfer the same to large Kjeldahl distilling flasks, add 100 cc of ammonia-free water, then about 2 grams of magnesium oxid in the form of milk of magnesia, and distill, collecting 150 to 200 cc of the distillate in 5 cc of standard hydrochloric acid. Continue the determination of nitrogen as usual.

TABLE I.—*Summary of results obtained in the determination of the nitrogenous constituents in cold-water extracts of meats.*

[Results expressed in percentages of the weights of fresh substance of meats.]

Laboratory No.	Kind of meat.	Composition of original material.															
					Nitrogen.												
							Soluble in cold water.										
		(1) Water.	(2) Fat.	(3) Ash.	(4) Total.	(5) Insoluble in cold water.	(6) Total.	(7) Coagulated by heat.	(8) Precipitated by zinc sulphate in filtrate from 7.	(9) Precipitated by bromin in filtrate from zinc sulphate in filtrate from 7.	(10) Proteid nitrogen (7+8+9).	(11) Non-proteid nitrogen.	(12) Precipitated by bromin directly.	(13) Precipitated by phosphotungstic acid in hot solution.	(14) Precipitated by tannin and salt.	(15) Precipitated by phosphotungstic acid in cold solution.	(16) As free ammonia.
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1649	Beef, sirloin, raw	75.46	3.08	1.02	3.40	2.66	0.74	0.29	0.06	0.02	0.37	0.37	0.29	0.18	0.38	0.37	....
1685	.....do	60.39	20.89	1.11	2.84	2.28	.56	.27	.02	.00	.29	.27	.25	.27	.26	.27	0.01
1667	Beef, neck, raw	71.29	8.77	1.02	3.10	2.47	.63	.34	.02	.01	.37	.26	.21	.29	.30	.32	.04
1671	Beef, flank, raw	59.17	24.95	.71	2.48	2.04	.44	.17	.01	.01	.19	.25	.19	.21	.23	.26	.01
1676	Beef, rump, raw	52.26	32.38	.74	2.40	1.94	.46	.22	.02	.00	.25	.21	.18	.22	.22	.23	.01
1677	Beef, round, raw	74.01	4.63	1.05	3.38	2.60	.78	.36	.03	.00	.39	.39	.25	.36	.38	.39	.02
1656	Veal, leg, raw	75.97	.96	1.15	3.47	2.62	.85	.40	.04	.01	.45	.40	.37	.39	.44	.42	.01
1662	.....do	75.53	3.99	1.10	3.23	2.58	.65	.26	.02	.02	.30	.35	.23	.24	.29	.25	.02
	Average, raw meats	68.01	12.46	.99	3.04	2.40	.64	.29	.03	.01	.33	.31	.25	.27	.31	.31	.02
1665	Beef, neck, boiled	54.40	13.68	.47	5.04	4.91	.13	.00	.02	.01	.03	.10	.00	.00	.03	.03	.01
1673	Beef, rump, boiled	38.35	42.03	.33	3.25	3.03	.21	.01	.04	.01	.06	.15	.01	.03	.07	.10	.02
1652	Veal, leg, boiled	64.73	1.59	1.01	5.31	4.88	.43	.01	.04	.02	.07	.36	.07	.06	.08	.10	....
1658	.....do	66.65	1.31	.75	5.36	5.06	.30	.02	.05	.01	.08	.22	.03	.01	.05	.06	.00

TABLE I.—*Summary of results obtained in the determination of the nitrogenous constituents in cold-water extracts of meats*—Continued.

Laboratory No.	Kind of meat.	Composition of original material.															
					Nitrogen.												
							Soluble in cold water.										
		(1) Water.	(2) Fat.	(3) Ash.	(4) Total.	(5) Insoluble in cold water.	(6) Total.	(7) Coagulated by heat.	(8) Precipitated by zinc sulphate in filtrate from 7.	(9) Precipitated by bromin in filtrate from zincsulphate in filtrate from 7.	(10) Proteid nitrogen (7+8+9).	(11) Non-proteid nitrogen.	(12) Precipitated by bromin directly.	(13) Precipitated by phosphotungstic acid in hot solution.	(14) Precipitated by tannin and salt.	(15) Precipitated by phosphotungstic acid in cold solution.	(16) As free ammonia.
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1658	Veal, leg, boiled	64.66	5.58	.59	4.80	4.61	.19	.00	.03	.02	.05	.14	.03	.02	.05	.06	.01
1664	....do	61.87	7.77	.66	4.90	4.67	.23	.01	.04	.01	.06	.17	.03	.03	.05	.03	.02
	Average, boiled meats	58.44	11.99	.64	4.78	4.53	.25	.01	.04	.01	.06	.19	.03	.02	.05	.06	.01
1674	Beef, rump, pan broiled	27.46	47.39	1.18	3.79	3.40	.39	.01	.03	.01	.05	.34	.02	.04	.02	.02	.02
1687	Beef, sirloin, gas broiled	48.47	23.70	1.33	4.36	3.84	.52	.04	.03	.01	.08	.44	.07	.08	.08	.10	.01
1688	....do	62.67	11.20	1.18	4.12	3.68	.48	.08	.02	.01	.11	.37	.09	.09	.09	.10	.02
1689	....do	70.23	7.45	1.02	3.61	3.05	.56	.17	.02	.00	.19	.37	.17	.18	.19	.20	.01
1659	Veal, leg, pan broiled	65.33	5.20	1.44	4.70	4.21	.49	.01	.04	.02	.07	.43	.04	.04	.03	.01	.02
1660	Veal, leg, roasted	68.35	4.65	1.36	4.22	3.72	.50	.01	.04	.02	.07	.43	.02	.03	.04	.03	.03
	Average for last six	57.09	16.60	1.25	4.13	3.64	.49	.05	.03	.01	.09	.40	.07	.08	.07	.08	.02
	Average for all determinations	61.86	13.56	.96	3.89	3.41	.48	.13	.03	.01	.18	.30	.13	.14	.16	.17	.01

(j) *Determination of nitrogen precipitated by phosphotungstic acid in the hot.*

Take 3 portions of 200 cc each. Transfer the same to 400 cc beakers (Schott and Genossen). Add 5 cc of 50 per cent sulphuric acid, stir thoroughly, and heat the solution to boiling on the iron plate. Now add drop by drop phosphotungstic acid solution until 1 drop gives no further precipitate in the clear supernatant liquid. Maintain the boiling temperature for about 5 minutes after precipitation is complete. Immediately filter the hot solution through a nitrogen-free filter, recently prepared with the use of boiling water. While filtering keep the liquid hot (90–100° C.). Wash the beaker, filter and precipitate thoroughly with hot water, having a temperature of 95–100°. Be careful the temperature of the solution and wash water is not less than 90° C. at any time. Determine the nitrogen in the precipitate produced by the phosphotungstic acid by the Kjeldahl method.

(k) *Determination of nitrogen precipitated by bromin directly in the cold water extract.*

Take 3 portions of 200 cc each. Transfer to Kjeldahl digestion flasks, acidify with 2 cc of normal hydrochloric acid, add about 2 cc of liquid bromin, and shake the contents of the flask very vigorously for some time (2 or 3 minutes at least). If the bromin is all taken up, add more until about 0.5 cc of liquid bromin is left undissolved and the supernatant liquid is thoroughly saturated with bromin. Allow the mixture to stand for 24 hours, decant the supernatant liquid through a double quantitative, nitrogen-free filter, and wash completely the flask, residue, and filter with water thoroughly saturated with bromin. Be sure the water is completely saturated with bromin. Return the filter and precipitate to the Kjeldahl flask and determine the nitrogen as usual.

(l) *Determination of nitrogen precipitated by bromin in the filtrate from the zinc sulphate precipitate.*

To the entire zinc sulphate filtrate add 2 cc of normal hydrochloric acid, dilute with an equal volume of water, and proceed with the determination as directed above in (k).

The results obtained are tabulated below in the two tables which follow. In Table No. 1 the results are expressed in percentages of the weight of the original meat. In Table No. 2 the results are expressed in percentages of total weight of nitrogen soluble in water. The data in both these tables were taken from tables in which the results had been calculated to the fourth place decimal, and for that reason there may in certain cases be apparently slight inaccuracies in the calculation as a result of condensing the same to the form in which they are here presented.

Table II.—*Summary of nitrogen determinations in cold-water extracts of meats.*

[Results expressed in percentages of total nitrogen soluble in cold water.]

Laboratory No.	Kind of meat.	Nitrogen.									
		(1) Coagulated by heat.	(2) Precipitated by zinc sulphate in filtrate from 1.	(3) Precipitated by bromin in filtrate from zinc sulphate.	(4) Proteid nitrogen. (1+2+3.)	(5) Nonproteid nitrogen.	(6) Precipitated by bromin directly.	(7) Precipitated by phosphotungstic acid in hot solution.	(8) Precipitated by tannin and salt.	(9) Precipitated by phosphotungstic acid in cold solution.	(10) As free ammonia.
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1649	Beef, sirloin, raw......	39.30	8.00	2.60	49.90	50.10	39.37	23.82	50.73	50.17	....
1685	.....do................	48.48	2.63	.68	51.79	48.21	44.13	47.64	54.15	48.43	1.11
1667	Beef, neck, raw	54.52	3.42	.83	58.77	41.23	39.16	48.15	47.54	51.15	6.54
1671	Beef, flank, raw	39.63	2.77	1.64	44.04	55.96	43.24	48.04	51.94	57.17	2.38
1676	Beef, rump, raw.......	48.97	4.39	.59	53.95	46.05	40.22	48.88	48.93	49.52	2.67
1677	Beef, round, raw......	46.14	3.44	.46	50.04	49.96	31.41	45.98	49.12	48.94	2.67
1656	Veal, leg, raw	46.36	5.11	1.40	52.87	47.13	43.33	45.48	51.68	49.03	1.57
1662	.....do................	40.66	3.66	2.29	46.62	53.38	34.71	36.14	44.56	38.97	3.06
	Average, raw meats..........	45.51	4.18	1.31	50.99	49.01	39.45	42.95	49.83	49.17	2.86
1665	Beef, neck, boiled....	1.58	15.42	5.60	22.60	77.40	13.53	8.16	24.34	23.05	8.99
1673	Beef, rump, boiled	4.63	19.84	4.30	28.77	71.23	4.86	13.23	33.44	48.09	8.50
1652	Veal, leg, boiled.......	3.22	9.06	4.13	16.41	83.59	17.23	14.71	18.08	23.37	....
1653	.....do................	5.61	16.10	4.22	25.93	74.07	8.73	2.76	15.14	20.35	1.32
1658	.....do................	1.26	14.92	10.24	26.42	73.58	14.44	11.03	25.32	31.25	2.57
1664	.....do................	3.18	15.55	4.01	22.68	77.37	13.00	11.37	19.91	14.68	9.43
	Average, boiled meats	3.24	15.15	5.42	23.79	76.21	11.97	10.21	22.71	26.79	6.16
1674	Beef, rump, pan broiled	2.22	7.18	1.88	11.28	88.72	6.33	10.05	.21	5.27	4.77
1687	Beef, sirloin, gas broiled	8.21	5.96	1.88	16.05	83.95	13.48	15.09	16.25	18.17	1.19
1688	.....do................	17.35	4.65	1.32	23.32	76.68	19.21	18.78	18.36	19.64	3.86
1689	.....do................	29.95	3.64	1.14	34.73	65.27	30.47	31.39	34.48	36.36	1.95
1659	Veal, leg, pan broiled..	2.69	7.49	3.56	13.72	86.28	8.12	8.25	5.25	1.70	4.98
1660	Veal, leg, roasted	.63	8.59	4.16	13.38	86.62	5.02	6.15	8.03	5.71	6.37
	Average for last six.............	10.17	6.25	2.32	18.74	81.26	13.77	14.95	13.76	14.47	3.85
	Average for all determinations	22.23	8.09	2.85	33.16	66.84	23.50	24.73	30.87	32.05	3.69

DISCUSSION OF RESULTS.

In general it may be said that while the triplicate determinations upon each sample agree fairly well, the results obtained with the different methods upon different samples of meats vary greatly. For example, in No. 1671 the nitrogen precipitated by bromin forms 98 per cent of the total proteid nitrogen, while in No. 1677 the nitrogen precipitated by bromin forms only about 63 per cent of the total proteid nitrogen. A comparison of the other methods show similar results. It does not seem probable that different cuts of meats or that meats from different animals would be so different in composition as to give such results. On the other hand, the several determinations have been made with the greatest precautions, and it does not seem possible at the present writing that such large variations can be due to the methods as now used. Further investigation of this subject is now under way, and it is hoped that the additional results which will soon be obtained will lead to a solution of this question.

(1) *Nitrogen coagulated by heat.*

In connection with the investigations which are being made in this laboratory it seems quite desirable to determine directly the amount of coaguable proteids contained in water extracts of meats. At first much difficulty was encountered in this determination in getting even fairly concordant results. In our work even a very slight excess of acetic acid has decidedly interfered with the complete separation and proper coagulation of the coagulable proteids. Under such conditions the proteid separates as a slimy film upon the surface of the liquid, the coagulation is not complete, and filtration takes place very slowly. In all such cases an additional separation takes place upon further evaporation of the filtrate for the precipitation of albumoses. In fact, separation of additional proteid continues until the solution is evaporated to dryness upon the water bath. This incomplete coagulation resulted even when 2 cc of N/10 acetic acid was used. Such difficulties were not so apparent in the analysis of cold-water extracts from cooked meats, which, however, contained as a rule very slight amounts of coaguable proteids.

In order to overcome these difficulties the coagulation in the later determinations was brought about in a neutral solution, after some preliminary experiments had proved conclusively that under such conditions the precipitation of coaguable matter was more complete, that the filtration under such conditions always took place readily, and that more concordant results were obtained. It was also proved that the coagulation was more complete when the solutions were made neutral to litmus paper than it was when the solutions were made neutral to phenolphthalein. This fact is shown by the following data:

TABLE III.—*Coagulation in neutral solutions.*

Solutions.	I.	II.
	Per cent.	*Per cent.*
Total nitrogen in water extract	0.765	0.755
Nitrogen coagulated in solution neutral to litmus paper	.303	.303
Nitrogen coagulated in solution neutral to phenolphthalein	.266	.269

In order to make the solutions from the water extracts of meats neutral to phenolphthalein it in all cases required the addition of 1 to 3 cc of N/10 sodium hydroxid more than it did to make the solutions neutral to litmus paper. Further studies are now being made upon this subject.

(2) *Nitrogen precipitated by bromin.*

The results reported in this connection confirm Bigelow and Harcourt's statement,[a] namely, "that the bromin precipitation from the original solution was found to hold only a small and variable portion of the proteids present." W. D. Bigelow,[b] also says "that more recent results in this laboratory indicate that bromin does not precipitate from aqueous solution all the proteoses and peptone present; at the same time, considering the small amount of these bodies contained in meat, it is believed that the results are approximately correct."

Van Slyke[c] and also Schjerning[d] have shown that bromin does not completely precipitate milk proteids and their derived caseoses and peptones.

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[a] J. Amer. Chem. Soc., 1901, **23**: 8, Proceedings.

[b] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 13 (part 10), p. 1396.

[c] Chem. News, 1903, **88**: 92.

[d] Zeit. anal. Chem., 1900, **39**: 545.

Fraps and Bizzell[a] concluded from a study of the application of the bromin method to the determination of vegetable proteids that bromin is not a suitable precipitant for proteids in vegetable materials.

It is evident from the results here presented that bromin does not even precipitate all of the proteid which is coagulated by heat in the case of raw meats. Assuming that the sum of the nitrogen coagulated by heat, precipitated by zinc sulphate, and precipitated by bromin in the filtrate from the zinc sulphate precipitate represents the total proteid nitrogen in the water extracts of meats, then the bromin precipitates 77.37 per cent of the proteid nitrogen in raw meats, 50.31 of the proteid nitrogen in boiled meats, and 73.48 per cent of the proteid nitrogen in broiled or roasted meats which is soluble in water.

(3) *Nitrogen precipitated by phosphotungstic acid in hot solution.*

The results obtained in this work show that phosphotungstic acid at a temperature between 90° to 98°, fails to precipitate all of the proteid material. In fact, it gives but slightly higher results than does bromin.

Mallet[b] proposed the use of phosphotungstic acid at 90° for precipitating proteids.

Fraps and Bizzell[c] found in their work that phosphotungstic acid did not precipitate vegetable proteids completely at 90° or 100°, but that this reagent at 60° precipitates very nearly the same quantity of nitrogen (with vegetable materials) as copper hydroxid.

(4) *Nitrogen precipitated by tannin and salt and nitrogen precipitated by phosphotungstic acid in the cold.*

The amount of nitrogen precipitated by these reagents in a number of cases are nearly alike, in other cases there is a wide difference in their precipitating power. The average results, however, show that in raw, broiled, and roasted meats they precipitate about equal amounts of nitrogenous substances, while in the water extracts of boiled meats, the phosphotungstic acid precipitates more than does the tannin and salt.

(5) *Nitrogen as free ammonia.*

The results show that there is a small amount of nitrogen as ammonia or ammonium salts.

The **President**. I believe there is also a paper on the separation of proteids in cereals to be presented.

DETERMINATION OF GLIADIN AND GLUTENIN IN FLOUR BY THE FLEURENT-MANGET METHOD.

By Joseph S. Chamberlain.

MOISTURE.

Moisture was determined in the air dry sample by the following method suggested by A. McGill, referee on cereal products:

> It is impractical, if not impossible, to dry flour and similar starchy material to constant weight below a temperature of 105° C. If exposed to air at this temperature for any considerable time, a notable increase of weight by oxidation occurs, especially in the presence of fats. For this reason an atmosphere of hydrogen or coal gas is necessary, and especially is this true if any component is to be estimated by difference, when the error in water determination enters into such estimation. The error due to the cause named may easily reach 2 or 3 per cent.

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[a] J. Amer. Chem. Soc., 1900, 22: 709.

[b] U. S. Dept. of Agr., Div. of Chem., Bul. No. 54.

[c] J. Amer. Chem. Soc., 1900, 22: 709.

On account of the convenience of coal gas this is generally used; and a very practical form of drying oven can be made by inserting a metal tube of 4-inch diameter through the oil bath used in hydrolysis of starch. This tube may have ends clamped on gas-tight and carrying nipples fitted with rubber tubing by which the dry gas is conveyed from the supply pipe. A tin tray to fit the bottom of the tube makes it easy to manipulate the samples to be dried; and these must be weighed in ordinary weighing bottles, or paired watch glasses, owing to the great hygroscopicity of the dry starch. Two hours' exposure at 105° C, using 2 grams of material and about 2 cubic feet of carefully dried coal gas per hour is sufficient for perfect desiccation.

Increase of weight through oxidation by air is very slow, indeed negligible for most purposes at temperature below 80°, so that the preliminary drying of the samples at this temperature may be effected in air, if desired.

Owing to the great hygroscopicity of dry starch, it is not recommended to weigh out from the dry sample. It is preferable to let the sample come into hygroscopic equilibrium with the air, and to keep it in carefully closed containers.

The results obtained by this method on the three samples of flour were as follows:

Serial No.	Moisture.
	Per cent.
6534......	13.79
6535......	12.25
6536......	12.77

All of the results in this report are determined on air-dry material and calculated to dry material on the basis of the above results for moisture.

THE FLEURENT-MANGET METHOD.

Fleurent[a] gives the following method for the determination of gliadin and glutenin in flour:

A potash solution containing from 3 to 3.5 grams of potassium hydroxid per liter is prepared with 70 per cent alcohol. The gluten from 33.4 grams flour is triturated in a mortar with portions of the alkaline solution, and the liquid transferred to a stoppered flask until 150 cc is made up. In the flask, to which some glass beads are added, the liquid is violently shaken during an hour until the gliadin is completely dissolved and the glutenin emulsified. On passing carbon dioxid into the liquid, the glutenin is precipitated, and the clear liquid, containing the gliadin, is (in aliquot part) dried at 105°, after filtration, and weighed. From this weight must be taken the weight of carbonate of potash in 50 cc of the solvent; the difference is the weight of dry gliadin, and can easily be calculated to a percentage on the flour. The glutenin per cent is determined by subtracting the gliadin from the total gluten, determined on a separate portion of the flour.

From extensive acquaintance with wheat flours of commerce, Fleurent gives the following limits: Glutenin, from 18 to 40 per cent gluten; gliadin, from 32 to 60 per cent gluten; and adds:

Bread made with a flour whose gluten contains as little as 20 per cent glutenin rises well during fermentation, but flattens in the baking. One always uses too much water with such a flour. When the gluten approaches 34 per cent of glutenin, the dough neither rises during fermentation nor in the oven, and the bread remains heavy and indigestible. Variations of 2 per cent in glutenin (from the normal 25 per cent) give rise to differences in the bread which are quite recognizable to the expert baker.[a]

Dr. Ch. Manget[b] suggests the following improvements upon Fleurent's process:

Alcohol of 71 per cent is employed because the water in the gluten reduces the strength to 70 per cent.

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[a] Manuel l'analyse chimique, 1898, pp. 308–314.

[b] Rev. intern. des falsif., 1902, p. 91.

The alcoholic potash solution is made as follows: Alcohol of 95 per cent at 15°, 775 cc; aqueous potassium hydroxid of 22 grams, per liter, 100 cc; distilled water, to make 1 liter.

In order to obtain a correction for the weight to be deducted from the gluten extract a mixture of 100 cc of the alcoholic potash with 50 cc alcohol (of 71 per cent) is saturated with carbon dioxid and one-third of the resulting solution evaporated to dryness at 105°. (Weight=R).

The determination is made by washing out the gluten from 30 grams of flour with a solution of chlorid of sodium (1 gram per liter). The separated gluten is air dried for one hour and weighed (moist gluten).

It is then shaken up with 100 cc of the alcoholic potash, in a stoppered bottle, furnished with glass beads, until a uniform emulsion is formed. This is transferred to a flask marked at 150 cc (or, better, specially graduated to allow for the volume of the moist gluten) and the washings of the bottle (71 per cent alcohol) are added, to make up to the mark. After very thorough agitation, 50 cc is poured out and dried, after supersaturation with carbon dioxid at 105°. The residue, less "R," gives dry gluten. The difference between this number and that previously found, gives moisture in moist gluten. The remainder of the solution is treated with carbon dioxid in excess, and 50 cc is filtered off and dried at 105°. The residue is gliadin from 10 grams flour (together with the weight R, which must be deducted). Glutenin is obtained by difference.

In the following work this method was followed, with two exceptions: (1) A 1 per cent sodium chlorid solution was used as wash water for determining gluten instead of a 0.1 per cent solution. This modification was introduced because it had been found from previous work that the more dilute the salt solution, the more gliadin was dissolved in washing and lost. (2) In obtaining the glutenin by subtraction Fleurent's original method was used and the gliadin subtracted from the weight of dry gluten dried at 105° to 110° instead of from the dry gluten as determined by Manget. At the same time that aliquot portions of the gliadin-containing filtrate were taken for the determination just described, similar portions were taken for nitrogen determinations, which were made by the Gunning method by T. C. Trescot, of the Bureau of Chemistry. The results obtained for gliadin and nitrogen are given in Table I:

TABLE I.—*Comparison of gliadin by weight and by nitrogen determinations.*

[On dry flours.]

Sample number.	Description of sample.	Gliadin.			Nitrogen in gliadin.
		By Fleurent's method (weight).	By nitrogen determination.		
			N×5.68.	N×6.25.	
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
6534	"Straight" flour from Kansas hard winter wheat.	8.77	7.78	8.56	16.43
		8.60	7.52	8.27	
6535	"Straight" flour from imported macaroni wheat.	12.72	11.42	12.56	16.18
		12.44	11.30	12.25	
6536	"Straight" flour from North Dakota macaroni wheat	11.84	10.68	11.75	15.97
		11.83	10.56	11.62	
		11.95	....	....	
		11.97	....	....	

It will be seen that the results from the nitrogen determinations agree quite closely with those obtained gravimetrically if the factor 6.25 is used. This fact is apparently substantiated by the determination of the percentage of nitrogen in the dry gliadin determined by Fleurent's method. The gliadin residues were washed into a Kjeldahl flask and the nitrogen determined with the results given in the last column of Table 1. These results agree quite closely with the theoretical 16 per cent of nitrogen corresponding to the proteid factor 6.25, but do not agree with the per cent 17.60

corresponding to the factor 5.68. It must be said, however, that there is no evidence that the weight of gliadin taken, upon which the per cents are figured, represents pure proteid. On the contrary, the probabilities are that the residue, after allowing for the potassium carbonate formed in the neutralization of the alcoholic potash solution, does not consist of perfectly pure gliadin.

Osborne and Voorhees[a] give 17.66 per cent of nitrogen in gliadin and 17.49 per cent in glutenin, purified as perfectly as possible, and use 5.68 as the factor for the two corresponding to a nitrogen content of 17.60 per cent. These results, if accepted as correct, show that the gliadin as determined by Fleurent's method is not pure proteid, although it is more nearly so than the gluten itself, as is shown by succeeding analyses. That this is so, and also that the gliadin as obtained by Fleurent's method is not the gliadin as it exists in the wheat, is further indicated by the following results.

By the method as described, as well as in many analyses of wheat and flour made by other methods, the gliadin and glutenin are determined in the gluten as obtained by washing out in water or dilute salt solutions. The following facts, bearing upon the question of the composition of gluten and its relation to the proteids of flour, were obtained by washing out the gluten in a slow stream of a 1 per cent sodium chlorid solution and then determining the percentage of nitrogen in the dry gluten. These results are given in Table II.

TABLE II.—*The composition of gluten in relation to the proteids of flour, with special reference to the effect of the 1 per cent sodium chlorid washing solution.*

[Calculated to dry flour.]

Sample number.	(1) Gluten. (By washing in 1 per cent NaCl solution.)	(2) Nitrogen, in gluten.	(3) True gluten. (N. in gluten ×5.68.)	(4) Nonproteid in gluten. (1−3.)	(5) Total proteid in flour. (N×5.68.)	(6) Proteid lost in washing. (5−3.)	(7) Proteid soluble in 1 per cent NaCl solution.	(8) True proteid. (3+7.)
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
6534	15.10	13.63	11.64	3.46	14.43	2.79	3.07	14.71
	14.61	13.24	10.96	3.65	14.37	3.41	3.29	14.25
6535	18.40	12.80	13.40	5.00	17.83	4.43	4.09	17.49
	17.60		12.78	4.82	17.72	4.94	4.43	17.21
6536	15.48	13.60	11.93	3.55	16.07	4.14	4.15	16.08
	15.37	13.59	11.87	3.50	16.06	4.19	3.69	15.56

On the glutens as given in column 1 the percentage of nitrogen was determined by the Gunning method, with the results given in column 2. These figures show that dry gluten, as ordinarily obtained and weighed, and on which the results in Fleurent's method are based, contains several per cent of nonproteid material. From the nitrogen determinations are obtained the amounts of proteid actually present in the gluten which are given as per cents of dry flour in column 3 of the above table. By subtracting these results from the gluten as weighed the amount of nonproteid material present in the gluten is obtained as given in the following column. These figures represent the material present with the gluten and considered as such, which should not be included, as it is not proteid. Consequently if the results for gliadin and glutenin are determined on this basis they can not be correct.

Again, from the true gluten determination, it is seen that not only does the gluten as washed out contain nonproteid material but the amount of proteid found in the gluten is less by several per cent than the total proteid contents of the flours. The difference between the total proteids found by nitrogen determination and the true gluten, or the amount of real proteid present in the gluten, is shown in column 6 of

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[a] Amer. Chem. J., 15: 469.

Table II, and indicates that there is a loss of from 2.79 to 4.94 per cent of proteids in the washing out of the gluten with a 1 per cent sodium chlorid solution. This result was to be expected, as a certain amount of the proteids of flour is known to be readily soluble in dilute salt solutions. In column 7 is given the percentage of the proteids found to be soluble by extraction in a 1 per cent sodium chlorid solution, and these figures agree in general with those given in the preceding column. If to the true gluten is added the amount of proteid soluble in the washing solution the total proteid present in the flour is obtained. These figures, as given in the last column of the table, should agree in general with the results for total proteids based on nitrogen determinations as given in column 5.

The determination of gliadin and glutenin, therefore, by Fleurent's method, using the gluten obtained by washing out with a 1 per cent sodium chlorid solution, does not give accurate results unless corrections are made both for the nonproteid material contained in the gluten and for the loss in washing of all, or nearly all, of the proteids soluble in a 1 per cent sodium chlorid solution. It remains to show whether or not the soluble proteid obtained from the gluten by solution in alcoholic potash and the subsequent precipitation of the insoluble proteid by neutralization with carbon dioxid is in reality gliadin, or whether with the gliadin other proteids are included.

EXTRACTION OF FLOUR WITH SALT SOLUTIONS AND WITH 70 PER CENT ALCOHOL.

As previously stated it was found that the more dilute the salt solution used for extraction the more gliadin or alcohol-soluble proteid was dissolved. By extracting the air-dry flour with salt solutions of varying strengths the following results were obtained:

TABLE III.—*Average amounts of proteid extracted by salt solutions of varying strength.*

[Calculated to dry flour.]

Sample number.	Sodium chlorid.			Potassium sulphate, 5 per cent solution.
	1 per cent solution.	5 per cent solution.	10 per cent solution.	
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
6534	3.29	2.73	2.20	2.16
6535	4.43	3.46	3.36	3.29
6536	4.15	3.52	2.83	2.73

It is thus shown that a 5 per cent solution of potassium sulphate extracts practically the same amount of proteids as a 10 per cent solution of sodium chlorid. Osborne and Voorhees used 10 per cent sodium chlorid in their work, but as it was necessary to determine the nitrogen in the filtrate a 5 per cent potassium sulphate solution was adopted as preferable because it gives off no hydrochloric-acid gas during digestion. The difference between the proteids extracted with 1 per cent sodium chlorid and 5 per cent potassium sulphate represents the extra amount of proteid, probably gliadin, extracted by the 1 per cent sodium chlorid.

By extracting air-dry flour with 70 per cent alcohol (sp. gr., 0.89) for varying lengths of time and at different temperatures, it was found that extraction for from eighteen to forty-eight hours at a temperature of from 25° to 30° C. yielded the maximum amount of soluble proteid. Three extractions each for a total additional time of seventy-two hours yielded only 0.23, 0.22, and 0.20 per cent increase, respectively, of soluble proteid. It is therefore believed that a simple extraction by standing, after a thorough shaking, for eighteen to twenty-four hours, or, better, a vigorous shaking for six to eight hours, dissolves practically all of the proteids soluble in dilute alcohol. The results obtained for soluble and insoluble proteids in the 70 per cent alcohol solution are given in the first two columns of Table IV.

TABLE IV.—*Extraction with 70 per cent alcohol and 5 per cent potassium sulphate.*

[Calculated to dry flour.]

Sample number.	(1) Proteid extracted by 70 per cent alcohol (18-48 hrs.).	(2) Proteid insoluble in 70 per cent alcohol.	(3) Proteid extracted from insoluble residue by 5 per cent solution K_2SO_4.	(4) Proteid extracted directly from flour by 5 per cent solution K_2SO_4.	(5) Proteids in alcohol extract soluble in salt solution (4-3).	(6) Glutenin (2-3).	(7) Gliadin (1-5).
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
6534	8.12	6.71	0.45	2.22	1.77	6.26	6.35
	8.18	6.47		2.16	1.71	6.02	6.47
6535	10.22	7.55	0.45	3.29	2.84	7.10	7.38
	10.28	7.66		3.46	3.01	7.21	7.27
6536	9.26	7.04	0.57	2.73	2.16	6.47	7.10
	9.03	7.34		2.78	2.21	6.77	6.82

It was found by extracting the insoluble residue from the alcohol extraction with 5 per cent potassium sulphate solution that only very small amounts of proteids were obtained, amounting to about 20 per cent of the total proteids soluble in that reagent. These results are given in column 3 of the above table. An average of nineteen other determinations of the same factor gave 0.60 per cent of the dry flour. This shows that the larger part of the proteids soluble in a 5 per cent potassium sulphate solution is removed when flour is extracted with 70 per cent alcohol. The residue from the alcohol extract, as given in column 2, will consist, therefore, of the glutenin plus the small amount of proteids soluble in salt solutions which has not been extracted by the alcohol. Subtracting from this insoluble residue the amount of proteids extracted from the residue by 5 per cent potassium sulphate the glutenin in the flour is obtained as given in column 6. The percentage of proteids found to be soluble in salt solution is given in column 4, while the figures in column 5 represent the amount of proteids included in the alcohol extract that are soluble in salt solution. The results given in column 7 are obtained by subtracting the figures in column 5 from the alcohol soluble proteids as given in column 1 and represent gliadin, the proteid soluble in 70 per cent alcohol, as described by Osborne and Voorhees.

DISCUSSION OF RESULTS.

In the following table the proteids as determined by the several methods and modifications are brought together for comparison with each other, and with the results given in Table IV:

TABLE V.—*Comparison of proteid determinations by various methods.*

[Calculated to dry flour.]

Sample number.	(1) Extraction method. (Sum of proteids.)	(2) By nitrogen determination. (Total proteids.) (N×5.68.)	Fleurent's method. (3) Gliadin.	Fleurent's method. (4) Glutenin.	Fleurent's method corrected. (5) True gliadin.	Fleurent's method corrected. (6) True glutenin.	Fleurent's method corrected. (7) Proteids soluble in 1 per cent sodium chlorid solution.	Fleurent's method corrected. (8) Sum of true proteids. (5+6+7.)
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
6534	14.83	14.43	8.77	6.33	7.78	3.86	3.07	14.71
	14.65	14.37	8.60	6.01	7.52	3.44	3.29	14.25
6535	17.77	17.83	12.72	5.68	11.42	1.98	4.09	17.49
	17.94	17.72	12.44	5.16	11.30	1.48	4.43	17.21
6536	16.30	16.07	11.83	3.65	10.68	1.25	4.15	16.08
	16.37	16.06	11.95	3.42	10.56	1.31	3.69	15.56

In column 1 of Table V is given the sum of the three classes of proteids named in columns 4, 6, and 7, of Table IV, determined according to the writer's method, while in column 2 is given the total proteids as calculated from the nitrogen determined by the Gunning method. In columns 3 and 4 are the gliadin and glutenin as determined by Fleurent's method, and column 5 and 6 the same figures corrected as previously described. In column 7 are given the proteid losses due to washing with a 1 per cent solution of sodium chlorid, and in the last column the sum of the true proteids obtained by Fleurent's method as corrected.

Comparing in the first place the totals obtained by the two methods as given in columns 1 and 8 with that given by the determination of nitrogen in column 2, it is seen that the sum of the proteids by Fleurent's method agrees with the latter nearly as well as does the sum by the extraction method. But comparing the amounts of gliadin and glutenin as determined by the two methods (Table V, columns 3 and 4, and Table IV, columns 6 and 7) there is seen to be a very great difference. The question at once arises—which is the true gliadin and glutenin? That the direct alcohol extract of the flour has extracted all of the alcohol-soluble proteid is shown by the preceding figures giving the amounts of proteid removed after 24 hours extraction by repeated extractions for 72 hours longer, which show that after 24 hours practically no more proteid is removed by 70 per cent alcohol. Therefore all of the gliadin must be in the alcohol-soluble extract.

That most of the proteids soluble in salt solution are also included in the alcohol extract is shown by the amounts of salt-soluble proteids left in the residue after alcohol extraction (Table IV, column 3). With this correction the alcohol extract represents all the alcohol-soluble proteid, and only that, and is therefore gliadin.

On the other hand, in the Fleurent method the gluten is first washed out from the flour, and this treatment, while it probably removes all of the proteids soluble in salt solution, also takes out a considerable portion of gliadin. More important than this is the fact that the gliadin is extracted from the gluten by dissolving or emulsifying the entire gluten with a 0.2 to 0.3 per cent alcoholic potash solution. While this is a very weak alkaline solution, still, as alkalies definitely affect the properties of proteids, it is at least questionable whether or not this treatment materially alters the solubility of these two proteids.

It seems that the Fleurent method yields too high results for gliadin and consequently too low results for glutenin. Also that the results based upon the gluten determination can not be correct because of the impure condition of the gluten. This error is not corrected by the Manget modification of Fleurent's method, as that simply gives a new procedure for determining the weight of dry gluten, without considering whether or not it is pure proteid.

PROPOSED METHOD.

In consideration of the results given above the following method, based upon the work of Osborne and Voorhees[a] and of Snyder,[b] is suggested to be used in comparison with other methods for the determination of proteids in flour:

(1) *Moisture.*—Determine moisture at 105° C. in an atmosphere of hydrogen or coal gas.

(2) *Physical condition of sample.*—Make all determinations upon air-dry flour, which should be either standard milled flour or, if laboratory prepared, capable of passing through bolting cloth 90 or 100 meshes to the inch.

(3) *Determination of proteids soluble in dilute salt solutions.*—Use a 5 per cent solution of potassium sulphate. Place 4 to 6 grams of the flour in a 150 cc or 200 cc flask and introduce exactly 100 cc of the salt solution. Shake thoroughly and allow to stand, with frequent shakings, for from 18 to 24 hours, or, better, still, agitate in a shaker for

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[a] Amer. Chem. J., 15: 469.

[b] Minn. Exp. Sta. Bul. 63, p. 519, 1899.

6 hours. After standing to settle, filter off 50 cc of the liquid and determine the nitrogen therein by the Gunning method. The amount found multiplied by 2 gives the nitrogen in the original flour. This result multiplied by 5.68 gives the amount of proteids in the flour soluble in a 5 per cent potassium sulphate solution.

(4) *Determination of alcohol-soluble proteid.*—Treat from 2 to 4 grams of flour in a similar way with 100 cc of 70 per cent alcohol, except that the extraction is made in a regular Kjeldahl digestion flask. The amount of alcohol need not, however, be exact, as the entire filtrate is used. After shaking allow to stand for from 18 to 24 hours, or, better, shake for from 6 to 8 hours. Allow the liquid to settle, filter, and wash the residue several times onto the filter paper, washing the whole thoroughly with alcohol. This is readily accomplished as the filtration is very rapid. Place the entire filtrate in a Kjeldahl flask and after strongly acidifying with sulphuric acid evaporate the filtrate until fumes appear and then determine the nitrogen by the Gunning method. Nitrogen × 5.68 equals the proteid soluble in 70 per cent alcohol.

(5) *Determination of proteids in insoluble residue from the alcohol extract.*—Return the filter paper and residue from the alcohol extract to the Kjeldahl flask and determine the nitrogen. Nitrogen × 5.68 equals the proteid insoluble in 70 per cent alcohol.

(6) *Determination of proteids extracted from the insoluble residue by 5 per cent potassium sulphate solution.*—Prepare a second alcohol extract and after returning the residue to the flask (a small flask may have been used in this case) extract as in (3) with a 5 per cent potassium sulphate solution and determine nitrogen in the extract. Nitrogen × 5.68 equals the proteids soluble in salt solution included in the insoluble residue.

(7) *Results.*—Subtract the proteids obtained in (6) from those obtained in (5) and the result is glutenin.

Subtract the difference between the proteids obtained in (3) and (6) from the proteids obtained in (4) and the result is gliadin.

Proteids obtained in (3) are the proteids soluble in salt solutions.

The **President**. If there is no discussion of the paper just read, the report on potash will be next received.

REPORT ON POTASH.

By F. B. Carpenter, *Referee.*

In accordance with the instructions of the association at the last meeting, your reporters have confined their study to securing a method which will recover all the potash in mixed fertilizers and to the determination of moisture in potash salts.

For this work three samples were prepared, one for the determination of potash and two for the determination of moisture.

In the early part of June a circular letter was sent to about forty chemists likely to be interested in the work, asking their cooperation, and a favorable response was received from fifteen. The directions for the work were outlined in the following circular letter, which accompanied each set of samples sent to those who had expressed a willingness to cooperate:

Dear Sir: At the last meeting of the Association of Official Agricultural Chemists the referee was directed to conduct further experiments for the purpose of securing a method that will dissolve all the potash in mixed fertilizers, giving attention to the amount of material used and the amount of water used for making the solution; also, if he has sufficient time, to study methods for the determination of moisture in potash salts. For the special study of the above subjects three samples have been prepared:

No. 1. Mixture acid phosphate and muriate of potash.
No. 2. Commercial muriate of potash.
No. 3. Commercial kainit.

DIRECTIONS FOR THE WORK.

1. Thoroughly remix material for analysis before beginning the work.

2. Moisture:

(a) Determine on all samples by official method, drying No. 1 for 5 hours in a steam bath at 100° C., and Nos. 2 and 3 in an air bath at 130° C. to constant weight.

(b) Determine on Nos. 2 and 3 by the following method: Heat 5 grams material in a platinum crucible (40 cc capacity) for 10 minutes with a small flame at a dark red heat, cool, and weigh. Repeat until constant weight is obtained.

(c) On sample No. 3 determine same as (b), covering material with about 5 grams freshly ignited lime.

3. Potash sample No. 1:

(a) Determine by official Lindo-Gladding method for fertilizers, described on pages 21 and 22, bulletin No. 46, revised edition, U. S. Department of Agriculture, Bureau of Chemistry. In making the solution, boil slowly so as to prevent any material loss of water by evaporation. In case there is considerable loss, the volume should be maintained by boiling water.

(b) Same as (a) with the addition of 20 cc concentrated hydrochloric acid before boiling.

(c) Determine in acid solution (b) by optional method described on page 22, bulletin No. 46.

(d) Determine like (a) (b) and (c) using 5 grams material instead of 10. Boil with same amount of water and make up to 500 cc as usual.

(e) Determine by any new or original method which will tend to facilitate the work or improve the results of the present official method.

4. Citrate insoluble potash, sample No. 1.

(a) Determine by method employed for citrate insoluble phosphoric acid, described on page 13, bulletin No. 46. After washing the insoluble residue from the citrate digestion, transfer to a 200 cc flask, add 20 to 25 cc concentrated hydrochloric acid, and boil until the larger part of the acid has evaporated; cool, dilute with about 125 cc water, bring to a boil, precipitate with ammonia and ammonium oxalate, cool, make up to mark, etc.; determine potash in 100 cc by the regular Lindo-Gladding method.

5. Make blank determinations of all reagents, using amounts required in regular analysis.

6. Use Gooch crucibles for filtering the potassium platinic chlorid, dissolving with hot water after weighing the precipitate and reweigh the crucibles.

7. In reporting, please describe fully any modifications of methods proposed, paying special attention to amount of material and amount of water used in making the solution.

Yours, truly,

F. B. Carpenter, *Referee.*
M. G. Donk, *Associate Referee.*

Sample No. 1 was made up as follows:

	Per cent potash (K_2O).
Acid phosphate (23.21 pounds)	0.04
C. P. potassium chlorid (1.79 pounds)	63.00
Total (25 pounds)	4.548

Both water and acid solutions of the acid phosphate were carefully analyzed for potash, and the results were found to be the same in both cases. The percentage of potash in the C. P. potassium chlorid used in the above calculation was the average of a number of determinations made in the laboratory of the Virginia Carolina Chemical Company, together with results furnished by the associate referee.

After further work on the method for the determination of potash, it was thought desirable by your reporter, to ask those who already had samples, to test the method outlined in the following circular letter which was mailed October 31:

Dear Sir: In connection with the A. O. A. C. potash work, I would thank you to make a determination of potash in sample No. 1 by the following modification of the Lindo-Gladding method, and let me have your result as soon as possible.

Boil 10 grams material with usual amount of water, plus 5 cc hydrochloric acid. Neutralize with sodium hydrate instead of ammonia. Otherwise follow the original method. Be careful to use only sufficient sodium hydrate to neutralize the solution.

Yours, truly,

F. B. Carpenter, *Referee.*

Owing to the late date on which this letter was sent out, only a few responses were received. Eighteen analysts took part in the work, either in whole or in part, and 13 laboratories were represented. The following tables show the results, by various methods, as reported by individual chemists:

TABLE 1.—*Potash determinations in sample No. 1.*

(Calculated potash content, 4.548 per cent.)

Analysts.	Amount of sample.						Neutralize with sodium hydrate.		Citrate insoluble.
	Official method.		Lindo-Gladding with 20 cc hydrochloric acid.		Optional with 20 cc hydrochloric acid.				
	10 grams.	5 grams.	10 grams.	5 grams.	10 grams.	5 grams.	Water solution.	Water solution + 5 cc hydrochloric acid.	
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
F. C. Weber, U. S. Department Agriculture	4.24		4.56						
	4.22		4.59						
	4.20		4.60						
H. D. Haskins, Massachusetts station	4.29	4.37	4.51	4.47	4.22	4.28		4.75	0.009
F. W. Robison, Michigan station	4.33	4.64							
	4.18	4.35	4.59						.293
	4.23	4.37	4.51	4.84					.254
M. G. Donk, Florida agricultural department	4.18	4.37	4.35	4.25		[a]4.46			
	4.27	4.25	4.04	4.32		4.44		4.44	
	4.31	4.36	4.23			4.47			
	4.14		4.44			4.43			
T. R. Gough, Maryland station	[b]4.26	[c]4.03	[b]4.28	[c]4.07		[c]4.13			.06
	[b]4.26	[c]4.00	[b]4.30	[c]4.09		4.26			.06
H. H. Hanson, Maine station								[d]5.15	
	4.28	4.06	4.19	4.24	3.86	4.84		[d]5.88	
	4.29	4.44	4.20	4.06	4.44	4.48		[d]6.11	
W. H. Scherffius, Kentucky station	4.10							4.42	
W. G. Morrison, North Carolina station	4.49	4.49	4.58	4.54	[d]3.58	[d]3.86		4.60	.08
C. C. McDonnell, South Carolina station	[e]4.33	[e]4.42			[f]4.40	[f]4.41			
G. Clausen Friend, Union Abattoir Co., Baltimore	4.49	4.55	4.66	4.70	[g]4.55	[g]4.59			.20
A. W. Bosworth, Rhode Island station	[h]4.55	[h]4.43	[h]4.62	[h]4.79		[h]4.79			
H. H. Hurt, Virginia agricultural department	[h]4.42	[h]4.44	[h]4.51	[h]4.50	[h]4.48	[h]4.40		[h]4.68	.08
Virginia-Carolina Chemical Co., Richmond, Va.:									
Edward Ryland	4.30	4.27	4.38	4.37	4.22	4.25	4.37	4.56	.00
S. H. Sheib	4.32	4.34	4.31	4.39			4.31	4.48	
H. M. McCance	4.22	4.18	4.26	4.38					.00
S. M. Robertson	4.31	4.26	4.31	4.34					
Harper Dean	4.32	4.33	4.32	4.25					
F. B. Carpenter	4.28	4.30	4.39	4.42	4.31	4.35	4.40	4.54	
Average	4.29	4.33	4.43	4.39	4.31	4.43	4.36	4.56	.10

[a] Modified as stated elsewhere.
[b] Used 5 grams to 250.
[c] Used 2½ grams to 250.
[d] Omitted from average.
[e] Final washing with 95 per cent alcohol.
[f] Water solution, final washing with 95 per cent alcohol.
[g] Used Gladding wash.
[h] No blank determinations were made.

In Table II the figures directly over the percentages represent the time of heating, expressed in hours when the sample was dried in an oven, and in minutes when ignited. The ignition was usually divided into periods of 10 minutes each, with gradual loss to last weighing.

TABLE II.—*Moistures determined in samples Nos. 1, 2, and 3.*

Analysts.	Sample No. 1.		Sample No. 2.				Sample No. 3.				
	5 hours at 100°.	Constant weight at 100°.	5 hours at 130°.	Constant weight at 130°.	Ignited 10 minutes.	Ignited over 10 minutes.	5 hours at 130°.	Constant weight at 130°.	Ignited 10 minutes.	Ignited over 10 minutes.	Ignited with lime.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
F. C. Weber, U. S. Department Agriculture	......	25	......	30	......	40	......	......	......	30	......
	5.41	5.71	2.59	3.24	......	3.88	1.75	2.68	......	9.72	7.92
H. D. Haskins, Massachusetts station	6.24	......	......	2.58	......	3.70	......	1.83	......	9.72	7.51
F. W. Robison, Michigan station	......	24	......	......	......	......	......	......	......	......	......
	6.70	7.60	......	......	......	......	......	......	......	......	......
M. G. Donk, Florida agricultural department	6.09	......	......	3.09	3.78	......	......	2.87	9.79	......	8.67
T. R. Gough, Maryland station	6.12	......	......	2.91	......	3.92	......	3.82	......	10.29	......
H. H. Hanson, Maine station	......	......	......	2	13	18	......	2	......	21	30
	6.03	......	......	3.40	3.77	3.86	......	[a]6.84	......	9.34	7.17
W. H. Scherffius, Kentucky station	......	8½	......	7½	......	40	......	9½	......	60	......
	5.93	6.02	2.68	2.85	3.71	3.81	2.32	2.54	9.87	10.31	......
W. G. Morrison, North Carolina station	6.12	......	......	3.30	......	3.89	......	2.50	......	10.08	......
C. C. McDonnell, South Carolina station	6.17	......	......	......	......	......	......	......	......	......	......
G. Clausen Friend, Union Abattoir Co., Baltimore	6.35	......	......	2.60	......	3.79	......	2.06	......	9.39	8.28
A. W. Bosworth, Rhode Island station	6.00	......	......	3.22	......	3.79	......	2.97	......	9.71	6.49
H. H. Hurt, Virginia agricultural department	6.22	6.30	......	3.31	......	3.81	......	3.20	8.07	9.53	......
Virginia-Carolina Chemical Co., Richmond:											
Edward Ryland	......	......	......	......	......	30	......	......	......	50	......
	6.10	......	2.85	3.06	3.69	3.78	2.18	2.69	8.56	9.70	8.47
F. B. Carpenter	......	......	7	12	......	60	7	25	......	60	......
	6.11	......	2.98	3.24	3.81	3.98	2.11	2.64	9.78	10.16	8.29
Average	6.10	6.41	2.78	3.04	3.75	3.84	2.09	2.71	9.21	9.81	7.85

[a] Omitted from average.

COMMENTS BY ANALYSTS.

F. C. Weber.—In moisture determinations, method (a) two results are given, and under method (c) "per cent loss from lowest weight obtained." In determinations by method (a) I was unable to obtain a constant weight after 25–30 hours, the loss in weight varying from 1 to 4 mg in sample No. 1, from 5 to 10 mg with No. 2, and from 1 to 8 mg with sample No. 3, this variation being least in the one gram samples. With method (c), sample No. 3, the material lost about 350 mg when it reached a point where it began to gain in weight, even if heated for 30 minutes and weighed as soon as cool. The gain in weight was always proportionate to time left in desiccator. The desiccators used were newly prepared from fresh calcium chlorid.

Floyd W. Robison.—In making the solutions the flasks were connected by reflux condensers, heated to boiling, and kept boiling for exactly 30 minutes.

M. G. Donk.—Determined moisture in samples Nos. 2 and 3 by simple ignition. The percentages are based on the loss after the first 10 minutes, because after the fifth ignition the weight was not yet constant, as shown by the following figures:

Loss of weight after repeated ignitions.

Time of ignition.	Sample No. 2.	Sample No. 3.
Weight after—	*Grams.*	*Grams.*
First 10 minutes	23.6500	23.6945
Second 10 minutes	23.6490	23.6898
Third 10 minutes	23.6475	23.6858
Fourth 10 minutes	23.6457	23.6835
Fifth 10 minutes	23.6445	23.6797
Sixth 10 minutes	23.6430	....

All possible pains were taken to heat to the same temperature each time, and at no time was a dull redness exceeded. With the muriate of potash, I can not say what causes the continued loss, but with the kainit decomposition of the salt and liberation of sulphuric acid seems to take place, as the ignited residue is alkaline.

Ignition of the kainit with lime was also unsatisfactory, for here the weight increased after the first ignition. The following are the figures of three successive ignitions of 10 minutes each:

Weight after first 10 minutes	29.0308
Weight after second 10 minutes	29.0435
Weight after third 10 minutes	29.0558

The loss after the first 10 minutes was used to calculate the per cent of moisture.

A portion of lime, ignited for several hours at a red heat, was heated in a covered platinum crucible for four successive 10-minute periods, then at a red heat for 10 minutes, next at a dull red for 3 hours, and another 3 hours at a red heat, giving the following weights in grams: 33.696, 33.846, 33.714, 33.762, 33.607, 33.703, 33.628.

It appears, therefore, that the ignition for moisture at dull redness is the temperature at which calcium oxid recombines with carbon dioxid. The variations between the figures for potash by solution with hydrochloric acid I attribute altogether to the large quantity of ammonium chlorid introduced by neutralization of the acid for precipitation, the presence of so much of this salt causing unavoidable loss by decrepitation and spattering on ignition with sulphuric acid. To overcome this I used the following modification of the optional method as used last year:

To the hot acid solution was added hot concentrated barium hydrate solution in slight excess, barely neutralized with ammonia, and precipitated lime and excess of barium with ammonium carbonate, cooled, made up to mark, and filtered. Fifty cc of the solution were evaporated to small bulk in a dish, transferred to platinum crucible, evaporated to dryness, ignited (with cover on crucible) at dull redness, dissolved residue in hot water, filtered, and the analysis completed as usual.

The use of barium hydrate introduces a minimum of ammonium chlorid, and ignition in a covered crucible avoids the possibility of mechanical loss. * * * In the sodium hydrate method the sodium introduced was not troublesome.

W. H. Scherffius.—I used a water oven for drying at 100° and an oil oven for 130°. In the latter case, one gram of material was used.

By the ignition method on sample No. 2 gradual loss was experienced for four successive ignitions of 10 minutes each, and on sample No. 3 the same loss was noted for six ignitions.

An experiment was made by drying 1 gram each of samples Nos. 2 and 3 in watch glasses in the oil oven at 120° C. The following results were obtained:

Loss in weight by drying 1 gram of sample at 120° C.

Time of heating.	Loss in weight.			
	Sample No. 2.		Sample No. 3.	
	Gram.	Per cent.	Gram.	Per cent.
5 hours	0.0259	2.59	0.0207	2.07
6½ hours	.0270	2.70	.0208	2.08
8½ hours	.0270	2.70	....	....

Instead of Gooch crucibles we use a form of the Fresenius filtering tube, modified by having the top expanded into the shape of a small funnel. In these tubes the precipitates can be thoroughly washed with less liquid than is required for a larger filter.

The ammonium chlorid wash in 6 or 7 successive portions of about 3 cc each is used instead of 5 or 6 of 10 cc, making the total amount used about 15 or 20 cc instead of 50 or 60 cc.

Determinations of potash on the two salts gave results as follows: No. 2, 48.88 per cent; No. 3, 12.66 per cent.

A. M. Peter.—The sodium hydrate method seems to afford a way out of the difficulty, provided the hydrochloric acid does not bring into solution more potash than is really water soluble. We must not lose sight of the fact that we are trying to determine water soluble potash, not total potash or acid soluble potash.

H. H. Hansen.—The moisture determinations on sample No. 3 were unsatisfactory either by the ignition method or drying at 130°. When heated with lime for less than 30 minutes the weight continued to decrease. When heated beyond this time the weight increased.

A. W. Bosworth.—There is an objection to the sodium-hydrate method on account of the very large amount of platinic chlorid necessary to satisfy the sodium chlorid present.

W. G. Morrison.—The optional method gives low results. It requires too many filterings, and unless the filtrates are large it is impossible to get all the potash. With an acid solution the amount of ammonium chlorid present becomes so great as to interfere with the work. The use of sodium hydroxid instead of ammonium hydroxid seems to offer no advantage, and the sodium residue with the platinum precipitate makes the process of washing much more troublesome. I prefer the official Lindo-Gladding method to any of its modifications.

DISCUSSION OF THE RESULTS.

It has been known by the association for some time that the official method gives low results on certain classes of mixed goods. Your referees have therefore endeavored to suggest changes that might tend to recover all the available potash present.

While we have not been altogether successful, it is hoped that some suggestions can be made which will be of assistance in future work.

In Table No. 1 it will be noted that the results by the official method, with one or two exceptions, are fairly concordant, both when 5 and 10 grams of material were used, but the percentages are in most cases considerably lower than theory. The ratio of the material to the amount of water used in making the solutions seemed to have no influence on the results. The acid modification of the official method tends to give higher percentages of potash, but the results are not altogether satisfactory. The introduction of such a large amount of ammonium chlorid appears to make the method more difficult of manipulation, and while it is believed that the addition of acid is necessary to liberate all the potash, there are other sources of error which partly compensate for what has been gained in this direction. The experience of the referee has been that while there was invariably a tendency to higher results, the results were always lower than theory and less uniform than by the official method.

From later work it appears to be unnecessary to use so much acid, in which case one of the objections mentioned will be partly eliminated and the results improved. The precipitation with ammonium and ammonium oxalate, however, seems to have the effect of holding back a small percentage of the potash. Knowing that filter paper has the tendency to absorb certain salts, experiments were made to see if any excess of potash was retained by the paper when solutions were filtered; the results, however, were the same, either by filtering or decantation. The acid modification of the optional method is altogether too troublesome to be considered as a possible method in fertilizer work.

Considerable time has been spent by the referee in trying to locate the cause of the low results on potash and suggest some method by which the difficulty could be overcome.

An experiment was made on sample No. 1 as follows: 10 grams were boiled with 300 cc of water for one-half hour, neutralized with ammonia, ammonium oxalate added, and the residue filtered off and washed. This residue was again boiled with the same amount of water, filtered, and washed. An acid solution of the resulting residue showed 0.30 per cent potash. This was repeated with 5 grams of material with similar results.

From a knowledge of the composition of the sample used, it seems more probable that the potash was absorbed or occluded in the insoluble residue than that the results were due to the formation of an insoluble compound. At any rate the results on the citrate insoluble potash given in Table No. 1 indicate that practically all the potash is available for plant food. A great many modifications of the Lindo-Gladding method were tried, but there seemed to be no hope of success without the use of acid in making the solution. Before sending out the samples quite a number of determinations were made on acid solutions with some promise of improvement, but from our later work it seems that in most cases when the precipitation is made with ammonia and ammonium oxalate a small proportion of the potash is again absorbed in the precipitate.

Mr. Ryland suggested that instead of neutralizing the solution with ammonia, sodium hydrate be used. This was tried, and the results were more promising than any modification before suggested. It was thought that possibly the higher results obtained by this method were due to the admixture of soda, but several of the precipitates of potassium and platinic chlorid were dissolved and reprecipitated with no loss, showing that the additional weight was due to potash obtained from the solution. Blank determinations were made in all cases to allow for any potash that might be in the soda solution. The soda appears only to act as a substitute for the potash which is usually absorbed in the precipitate formed by the addition of ammonia and ammonium oxalate.

The principles of the method are: (1) Use a small amount of hydrochloric acid to liberate any occluded potash in the substance; (2) neutralize with soda instead of ammonia to prevent any occlusion or absorption of potash in the precipitate usually formed by the addition of ammonia and ammonium oxalate. It is best to use only a small amount of acid in making the solution (5 cc were found to be sufficient) so as to avoid using a large amount of sodium hydrate. It is convenient to add a little phenolphthalein as indicator, thus avoiding a large excess of the alkali.

Owing to the short time which has elapsed since this modification was proposed only a few results have been obtained. These are given in Table No. 1. Some of these figures are high, probably for the reason that all of the soda was not removed from the precipitate of potassium platinic chlorid, but from the experience of the referee it is believed that this difficulty could be readily overcome in practice.

The members of the association will recall the time when the Lindo-Gladding method required the addition of 5 cc of sodium chlorid solution to the solution of potassium chlorid, just previous to the precipitation with platinic chlorid. While in this proposed method the sodium chlorid is present for another purpose, the same conditions are obtained for precipitation as in the old Lindo-Gladding method. One analyst makes objection to the method on account of the very large amount of platinic chlorid required to satisfy the sodium chlorid present. Our experience is that no larger amount of platinum solution is required than in the official method. The potash is first satisfied, and the excess precipitates a part of the sodium chlorid which is readily soluble in 80 per cent alcohol, and the remaining excess of sodium chlorid is readily removed with Gladding wash. Not enough work has been done with the method as yet to form any definite conclusions, but the results are thus far very promising.

Mr. C. L. Hare, in his report on potash for 1901, referring to the milk of lime method, says: "While the direct evaporation of the solution with platinum solution leaves a rather large amount of lime salts, none of the analysts has reported trouble in the final washings. In washing this residue analysts who have little experience with the method are apt to subject it to too vigorous rubbing. Experience teaches that excessive pulverization is unnecessary and will possibly result in loss of potash caused by finely divided potassium platinic chlorid passing through the filter." It is the latter point which should be brought out in connection with any of the Lindo-Gladding modifications which have been suggested in this report. Experiments made by the referee show that the official method has a decided tendency to lower results if the precipitate is excessively pulverized, and this may account for some of the variations in results reported in Table I.

MOISTURE.

Sample No. 1. Acid phosphate and muriate of potash.—The results by the official method, with two exceptions, are fairly concordant. The samples were carefully sealed, and such differences as occur are undoubtedly due to manipulation after opening.

Sample No. 2. Muriate of potash.—By the official method the results are not satisfactory, there being a maximum variation of 0.72 per cent, with the highest result only slightly over 3 per cent.

By the ignition method the results are more uniform, but are materially higher than those by the official method. The difference is probably due to water of crystallization in the impurities, which is only slowly liberated at 130°.

Sample No. 3. Kainit.—The referee infers that if no explanation accompanied the reports the results at 130° represent percentages obtained by drying to constant weight. Our experience has been that kainit continues to lose weight for a long time at this temperature, and this undoubtedly accounts partly for the variation in results. One analyst reports a result of 6.84 per cent by drying for two hours at 130°, which is over 3 per cent higher than any other result. The kind of oven and the manner of inserting the thermometer undoubtedly has much to do with these variations. If this method is to be used, it would be well to specify the style of oven, and have a definite time of drying, rather than to specify "to constant weight."

The ignition method offers altogether different conditions. Here not only the mechanical moisture, but also the chemically combined water is liberated. There is also some decomposition, resulting in the loss of some of the volatile constituents. The use of lime to absorb the acids which have been liberated was not very satisfactory, as is shown by the results from different laboratories.

In using the lime method it was the experience of the referees, and several other analysts have reported the same difficulty, that after heating for a certain time the substance increases in weight. This is undoubtedly due to the absorption of carbonic acid. The method is very troublesome, and if it were not for the fact that this is practically the method used by German chemists, by which all settlements for kainit purchased from foreign producers must be made, no further consideration of the same would be recommended. It remains for this association to say whether the total water content of the salt shall be determined, or we shall adhere to the present method, with possibly some modifications, and attempt to approximate the mechanical moisture. It is possible that better results could be obtained with a somewhat lower temperature than 130°. Mr. Scherffius reports concordant results on samples Nos. 2 and 3 by drying in an oil oven for 6½ hours at 120°.

As has been suggested by several analysts, the percentage loss by the ignition method depends very much on the time of heating and the temperature employed. It is very difficult to specify conditions which will insure absolute uniformity in the hands of different analysts; the best that can be done is for each analyst to determine for himself what conditions will give him the most satisfactory results.

RECOMMENDATIONS.

Since the last meeting of the association nothing of especial value has been published on the determination of potash. Attention has, however, been called to the phosphomolybdic method, first elaborated by "Wavalet." The method as it stands presents no advantages over the one in present use, except that it eliminates an expensive reagent. If modified so as to be carried out volumetrically like the present volumetric phosphoric-acid method, it might prove a valuable addition to the official methods.

The results for this year do not justify any recommendation for a change in the official method for the determination of potash, but it is suggested that the modification requiring a slightly acid solution and neutralization with sodium hydrate be given further trial. The referee recommends that some action be taken to ascertain whether the members desire to determine moisture in potash salts by drying at a stated temperature or by ignition, and that the study of the method thought most desirable be continued with a view to getting more concordant results.

I wish to thank all those who have so willingly contributed their time to the work outlined by the referee and to make special mention of Mr. M. G. Donk, associate referee, and Mr. Edward Ryland, one of my assistants, who have rendered valuable assistance in the preliminary and experimental work.

The PRESIDENT. Is there any discussion on the points raised by the referee?

Mr. ROSS. Is the theory in regard to the use of the sodium hydroxid that the energies of the precipitate are directed toward an occlusion of the sodium salts rather than the potash salts?

Mr. **Carpenter.** That is the only explanation I can offer.

Mr. **Robinson.** Would it not be possible to use asbestos to carry down the precipitate mechanically, and keep it from creeping?

Mr. **Carpenter.** I do not see that there would be any objection if we knew that the asbestos had no potash in it.

Mr. **Cameron.** There is this objection: The majority of the samples of asbestos on the market are magnesia bearing, and the magnesia would probably precipitate much more of the platinum reagent.

Mr. **Williams.** Some time ago I made an analysis of asbestos which contained over 3 per cent of total iron. I did not determine whether it was ferrous or ferric.

Mr. **Huston.** In making determinations of moisture in my laboratory on mixtures of phosphates and potash salts it was found that fumes of hydrochloric acid were continually given off from a mixture of hydrochloric acid and muriate of potash, and accurate moisture determinations were exceedingly difficult to obtain. I presume the same difficulties would occur in dealing with sample No. 1. In fact, after working on the samples a while the copper of the baths was strongly attacked by the hydrochloric acid which was given off by the mixture of acid phosphate and only a small amount of muriate of potash, certainly less than was contained in sample No. 1.

The **President.** If there is no further discussion the report of the committee on nominations will be received.

REPORT OF COMMITTEE ON NOMINATIONS.

Mr. **Wheeler.** On behalf of the committee I place the following names in nomination: For president, Mr. M. E. Jaffa, of California; for vice-president, Mr. C. L. Penny, of Delaware; for secretary, Mr. H. W. Wiley, of Washington, D. C.; as additional members of the executive committee, Mr. W. P. Headden, of Colorado, and Mr. W. R. Perkins, of Mississippi.

Mr. **Van Slyke.** I move that the nominations be approved and the officers named be declared elected accordingly.

The motion was unanimously carried.

The **President.** The report on soils is now in order.

REPORT ON SOILS.

By F. P. Veitch, *Referee.*

Only two laboratories have participated in the work on soils. Mr. Seidell, of the Bureau of Soils, Department of Agriculture, made an examination of methods for the analyses of alkali soils; Mr. Moore and the referee worked on methods for determining available plant food, while the referee alone devoted a great deal of time to the study of acidity and methods for its determination. The cooperative work for 1903 was outlined in the following circular:

Outline for A. O. A. C. Work on Soils for 1903.

Owing to the fact that we have no methods which may be regarded as furnishing an accurate measure of the available plant food of a soil, it does not seem advisable to the referee to ask chemists to compare their results on a particular set of soil samples. For this and other reasons, not necessary to state here, it appears more rational to request those who can take part in the soil work to secure their samples from soils of which they know the crop history and fertilizer requirements, and endeavor to determine the available plant food in these samples by the methods mentioned below or by any other methods deemed worthy of trial. As the number of samples worked on by each chemist will be small, several methods may be tried without overburdening the individual. The results, together with the crop and fertilizer history of the soils, will be summarized by the referee and it is hoped to get comparisons of several methods on a large number of samples of known soils. It is particularly requested that those chemists dealing with soils which have never shown the need of fertilizers will cooperate heartily in the work in order that we may at least begin to establish standards with which to compare fertilized soils.

The lines of work upon which cooperation is requested are the following:

(1) Determination of available potash and phosphoric acid of virgin and cultivated soils in samples taken from each well-marked stratum of soil to the depth of four feet.

(2) Investigation of the most desirable amounts of soil and water to be used in dissolving the soluble salts of alkali soils, and of the time required for the establishment of equilibrium between the soil and the solvent.

(3) Investigation of methods for the estimation of soil acidity.

1.—DETERMINATION OF AVAILABLE POTASH AND PHOSPHORIC ACID.

The referee will furnish a sampling tube with which samples can be taken to a depth of 4 feet. The tube must be promptly returned to the Bureau of Chemistry, U. S. Department of Agriculture, or forwarded to another chemist, as may be directed, charges prepaid.

(a) Determination of potash dissolved by treatment with distilled water, following strictly the method of Moore,[a] except that distilled water is used as a solvent instead of N/200 hydrochloric acid.

It is not necessary to use acidified alcohol in washing the precipitate of potassium platinic chlorid, as ordinary 90 per cent alcohol serves the purpose just as well.

The referee has used this method on some 15 or 20 samples of soil and in all cases as much or more potash was obtained as was removed from the soil by the oat crop.

(b) Determination of available potash and phosphoric acid in N/200 hydrochloric.[a] Do not correct for basicity, but secure a uniform temperature of 40° C. and moderate agitation in Wagner machine.

The results secured last year on surface soils were not very satisfactory, but it is hoped that the estimation of the available material to greater depths will give better results.

2.—METHODS FOR THE ANALYSIS OF ALKALI SOILS.

(a) Treat 10 grams of soils representative of different classes of alkali with 1 liter of distilled water, shaking frequently, and determine bicarbonates, carbonates, chlorids, and sulphates, after standing 12 hours, 24 hours, 48 hours, and 72 hours.[b]

(b) Treat 10 grams, 25 grams, and 50 grams of the soils used in (a) with 1 liter of distilled water with frequent shaking for the time giving the greatest solubility in (a) and determine bicarbonates, carbonates, chlorids, and sulphates.

The referee would impress upon those members of the association who have to deal with alkali soils the importance of having in our methods processes for the satisfactory estimation of the soluble salts in these soils. He would further urge that it is the duty of those so situated to work out and propose methods for this purpose. The referee therefore makes a personal appeal to each member in the alkali regions to devote some attention to this line of work this year in order that methods may be adopted, provisionally at least, for such cases.

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[a] J. Amer. Chem. Soc., **24**: 79.

[b] U. S. Dep. Agr., Bureau of Soils, Bul. No. 18.

3.—METHODS FOR THE DETERMINATION OF SOIL ACIDITY.

(Use Jena glassware for all work.)

As it may be inconvenient for many to secure suitable samples for this work, the referee will furnish samples to those who request them.

A.—Determination of acidity by the sodium chlorid method proposed by Hopkins, Knox, and Pettit.[a]

In the execution of this method note the following points:

(1) Determine as far as possible whether or not there is any appreciable amount of free strong acid in the sodium chlorid filtrate from the soil. This may be done by comparing the reaction of the filtrate with 5 per cent sodium chlorid solution to which known amounts of strong acid have been added, together with an indicator, such as methyl orange, and noting the sharpness and strength of color in the two cases.

(2) Note whether or not the end point is sharp, and state whether the titration was stopped when a very faint permanent pink color was secured, or continued until the color was strong.

(3) Note whether or not a precipitate is produced on adding the alkali to the boiling liquid; and if so, filter it off after titration, wash with water and determine what bases it contains, and estimate each accurately.

(4) Express all results on the basis of the water-free soil in parts of lime per million of soil.

B.—Determination of acidity by the limewater method proposed by Veitch.[b]

C.—Determine the reaction of the original soil furnished by the method proposed by the referee.[a]

DETERMINATION OF AVAILABLE POTASH AND PHOSPHORIC ACID.

Available potash and phosphoric acid were determined by the N/200 acid method, and available potash by simple treatment with water.

Potash and phosphoric acid were determined in different soil layers in samples drawn from the fertilized plots of the Maryland station. This soil is deficient in available phosphoric acid. The referee was unable to obtain soils to different depths which are known to be deficient in potash.

Most of the soils which have been worked on are drawn from the collection of the Bureau of Chemistry. The amount of potash and phosphoric acid removed from the soils by various crops is accurately known, thus affording an absolute standard with which to compare analytical results. I desire to express my great indebtedness both to Mr. Wiley and Mr. Moore for samples of soil and permission to transcribe their records. Both analytical and crop data are given in Table I.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 73, Proceedings Nineteenth Annua Convention, A. O. A. C., 1902, p. 114.

[b] J. Amer. Chem. Soc., 1902, **24**: 1120; Proceedings Nineteenth Annual Convention, A. O. A. C., 1902, p. 136.

TABLE I.—*Plant food removed by oats, by digestion in distilled water, and in N/200 acid (C. C. Moore and F. P. Veitch).*

[Parts per million.]

Description of sample.	Removed by oats.		Water soluble.	N/200 acid soluble.	
	K_2O	P_2O_5	K_2O	K_2O	P_2O_5
Vegetation experiment, Bureau of Chemistry:					
California soil, pot No. 1	26.0	....	16.0	....	....
Illinois station soil, pot No. 5	57.0	....	17.0	....	....
Massachusetts station soil, pot No. 12	27.0	....	37.0	....	....
South Dakota station soil, pot No. 22	27.0	....	18.0	....	....
California cultivated soil, pot No. 63	14.0	....	21.0	....	....
Massachusetts cultivated soil, pot No. 78	34.0	....	22.0	....	....
Michigan cultivated soil, pot No. 80	17.0	....	17.0	....	....
Wisconsin cultivated soil, pot No. 90	28.0	....	16.0	....	....
Pennsylvania cultivated soil, pot No. 129	40.0	....	28.0	....	....
Cooperative Experiments, 1902:					
Wisconsin station soil (barnyard manure used)	38.0	35.0	27.0	30	64.0
Vermont station soil	22.0	17.0	82.0	56	1.5
Illinois station soil	34.0	9.0	33.0	31	2.0
Missouri station soil	30.0	7.0	34.0	42	2.0
Virginia station soil	62.0	31.0	137.0	217	18.0
Arlington station soil	15.0	3.0	20.0	41	2.0
North Carolina station soil	9.0	2.0	17.0	50	1.0
Florida station soil	7.0	4.0	12.0	20	5.0
Wyoming station soil	36.0	11.0	50.0	55	1.0
Rothamsted soils, barley continuously:					
Plot B_{1a}, only nitrogen applied	17.0	6.0	6.0	....	....
Plot B_{2a}, N and P_2O_5 applied	18.0	9.0	....	....	....
Plot B_{3a}, N and K_2O applied	49.0	8.0	53.0	....	....
Plot B_{4a}, complete fertilizer applied	45.0	14.0	40.0	....	....
Rothamsted soils, wheat continuously:					
W_3, unmanured continuously	14.0	5.0	10.5	....	....
W_{10a}, only nitrogen applied	14.0	4.0	7.0	....	....
W_{11}, N and P_2O_5 applied	19.0	10.0	3.5	....	....
W_{13}, complete fertilizer applied	45.0	14.0	44.0	....	....
Hemp experiments, Bureau of Chemistry, Kentucky soil	36.0	4.0	23.0	120	2.0
Do	70.0	27.0	44.0	98	14.0
A. O. A. C. samples:					
No. 1, Ohio station, lacks P_2O_5	....	....	12.0	55	.6
No. 2, Pennsylvania station, lacks P_2O_5	....	....	33.0	111	1.5
No. 3, Maryland station, lacks P_2O_5	....	....	104.0	142	10.0
No. 4, Maryland station, well supplied with all	....	....	40.0	42	4.3
No. 5, Massachusetts station, lacks P_2O_5	....	....	65.0	175	.7
No. 6, Kentucky station, lacks K_2O	....	....	4.0	18	4.3
No. 7, Rhode Island station, lacks K_2O	....	....	2.0	12	1.3
No. 8, Rhode Island station, lacks P_2O_5	....	....	2.0	42	1.0
Maryland P_2O_5 experiments:					
Plot No. 10, 0 to 8 inches, lacks P_2O_5	....	....	3.2	29	.7
Plot No. 10, 8 to 16 inches	....	....	3.0	13	.5
Plot No. 10, 16 to 36 inches	....	....	2.0	16	.7
Plot No. 11, 0 to 8 inches, well supplied with all	....	....	1.0	25	5.0
Plot No. 11, 8 to 16 inches, well supplied with all	....	....	1.0	12	.7
Plot No. 11, 16 to 36 inches, well supplied with all	....	....	1.0	17	.7
Maryland K_2O experiments:					
Plot No. 4, 0 to 8 inches, well supplied with all	....	....	22.0	100	1.5
Plot No. 4, 8 to 16 inches, well supplied with all	....	....	18.0	166	1.5
Plot No. 4, 16 to 36 inches, well supplied with all	....	....	22.0	180	1.0

From these results it appears that the N/200 acid method when not corrected for bacisity extracts less phosphoric acid from most soils than is removed by crops, but usually dissolves more potash than oats remove. Could it be assumed that plants always feed to considerable depth, say 3 or 4 feet, the low solubility frequently shown would be amply sufficient for maximum crops. However, we have positive data showing that from a given weight of soil the oats obtained more phosphoric acid than the method shows to be available.

From the fact that this method, like most others, distinguishes quite sharply between fertilized and unfertilized plots, the indications are that acid of this concentration attacks chiefly the lime phosphates, while iron and alumina phosphates, as well as the phosphorus of organic matter, is apparently but little affected. For these reasons it appears desirable to study the solvent effect of more concentrated acids, at the same time endeavoring to reach the phosphorus in organic combination. For the same reason it is imperative to correct for basicity of the soil, otherwise only the solubility of calcium phosphate in salt solutions is obtained. A method is now being worked out for estimating the free mineral acid in the presence of acid salts, and it is hoped that valuable data will be obtained along this line. It is to be borne in mind in devising a method for available phosphoric acid that the activity of fungi may be reasonably expected to constantly place more phosphoric acid within reach of the higher plants, and it is practically impossible to prognosticate how much may be thus rendered available or to devise a chemical method that will measure it.

The potash declared available by this method appears to be usually greater than that removed by the oat plant, even under the most favorable conditions, and is much greater than is usually believed to be necessary for the production of maximum crops under field conditions. As potash, relatively speaking, is seldom the controlling element in our soils, it can not be assumed that the quantities shown by the method are not actually available, for other factors limit the amount removed by the plant.

The results on water-soluble potash have not generally borne out the indications of preliminary work with this solvent. The results, however, have sufficient significance to justify further work along this line, changing the ratio of solvent to soil from 5:1 to 10:1. One difficulty with this solvent is that it is often impossible to obtain a perfectly clear filtrate, but it has been found that the addition of acid for the purpose of flocculating the clay in the filtrate dissolves inappreciable quantities of potash from the clay.

METHODS FOR THE ANALYSIS OF ALKALI SOIL.

The referee was unable to do any work on this subject, and at his special request Mr. Seidell and Mr. Brown, of the Bureau of Soils, U. S. Department of Agriculture, undertook a study of the ratio of soil to water and of the effect of time on the solubility of alkali salts. The results obtained were reported by Mr. Seidell as follows:

REPORT ON ANALYSIS OF ALKALI SOILS BY A. SEIDELL.

The soil samples employed represented three classes of "alkali," characterized, respectively, by an excess of sulphates, carbonates together with chlorids, and chlorids.

In the experiments planned to show the effect of time upon the amounts of salts dissolved (first three analyses of each sample), three portions of each soil sample were used, one portion being for the one-day digestion period, another for the two-day, and a third for the three-day period. The weighed 5-gram portions of each sample were placed in flasks and 500 cc of water added, after which all of the flasks were shaken thoroughly for several minutes. After standing 24 hours the first set of three solutions was analyzed. The analysis consisted in siphoning off about 400 cc of the cloudy solutions, using 100 cc portions for the titrations, and, after clearing the remainder by means of several drops of hydrochloric acid, 100 cc portions were taken for the determination of sulphates.

The second set of flasks, after having stood 48 hours without additional shaking, were treated in the same manner as the first set. The third set were shaken well at the end of 48 hours and then allowed to stand 24 hours longer before being analyzed.

From the results obtained as above described it appears that in some cases there is a slight increase in the amount of soluble salts obtained by longer digestion, while in other cases there is a slight decrease. It therefore appears that no advantage results by extending the time of digestion beyond one day. It is possible, however, that if the experiments were modified so that the solutions were shaken at frequent intervals, or shaken continually by means of a suitable apparatus for different lengths of time, the amounts of salts dissolved would bear a definite relation to the lengths of time of the digestion.

The results obtained from the analysis of the solutions prepared with varying amounts of soil to the same amount of water show, on the other hand, in most cases a somewhat less percentage of soluble salts than are found when the amount of water to each gram of soil is greater. This fact is also brought out by the results of the analyses of alkali soils made by Mr. Breazeale and Mr. Veitch, and given in the report[a] of the referee on soils for last year.

It is to be noticed, however, that with alkali soils containing relatively large amounts of soluble salts the differences obtained by the use of 10 grams of soil to 1 liter of water and of 50 grams of soil to 1 liter are proportionately less than the differences found for soils containing very small amounts of readily soluble material.

It will also be seen that while the results presented last year indicate that the sulphate determination is most affected by the concentration of the solution, the results given in the accompanying table show that the sulphate determination is affected no more by change in the proportion of soil to water used than is the determination of chlorin or carbonates.

On the whole, it appears that while the use of less than 50 grams of soil to 1 liter of water yields higher results in most cases, especially those where the percentage of "alkali" is low, it is probably no great advantage to use the larger amount of water, since by keeping to the proportions already adopted in the Bureau of Soils it is very certain that all of the soluble material to which the "alkali" qualities of the soil are to be attributed are brought into solution and easily determined by the ordinary analytical processes.

The use of less soil than 50 grams per liter will no doubt cause some inconvenience and diminish the accuracy of the work in cases where the soil contains very small amounts of alkali, and therefore yields a very dilute solution in which the several determinations are to be made. There is probably no very great advantage to be attained in the use of any one of the ratios of soil to water here considered, either in respect to the facility of performing the analytical operations or the character of the results obtained. The laboratory of the Bureau of Soils has used the ratio of 50 grams of soil to 1 liter of water for four years, and in that time made several thousand analyses of alkali soils from all the principal alkali areas of this country and several areas in foreign countries.

In illustration of the points brought out in this report I submit herewith the analytical results obtained by myself and Mr. Bailey E. Brown, working independently.

TABLE II.—*Results on maximum solubility of alkali soils, and effect of time and variation of relative proportion of soil to water.*

[Results expressed in percentage of air-dried soil.]

ANALYSES MADE BY A. SEIDELL.

SAMPLE NO. 6603. (BILLINGS, MONT. ALKALI CRUST. TOTAL SALTS BY ANALYSES, 2.58 PER CENT.)

Time in days.	Ratio.		CO_3.	HCO_3.	Cl.	SO_4.
	Soil.	Water.				
	Grams.	*cc.*				
1	10	1,000	0.00	0.105	0.246	1.65
2	10	1,000	.00	.105	.246	1.64
3	10	1,000	.00	.099	.228	2.01
1	25	1,000	.00	.06	.14	1.6
1	50	1,000	.00	.05	.13	1.6
[a]1	50	1,000	.00	.03	.084	1.63

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[a] Proceedings Nineteenth Convention of the A. O. A. C., 1902, p. 112.

Table II.—*Results on maximum solubility of alkali soils, and effect of time and variation of relative proportion of soil to water*—Continued.

SAMPLE NO. 6678. (WALLA WALLA COUNTY, WASH., ALKALI CRUST. EFFERVESCED WITH HYDROCHLORIC ACID. TOTAL SALTS, 22.54 PER CENT.)

Time in days.	Ratio.		CO_3.	HCO_3.	Cl.	SO_4.
	Soil.	Water.				
	Grams.	*cc.*				
1	10	1,000	2.918	3.15	11.26	2.25
2	10	1,000	3.57	2.12	11.47	2.49
3	10	1,000	3.27	2.72	11.50	2.60
1	25	1,000	2.86	3.51	11.47	2.37
1	50	1,000	3.34	2.06	10.69	2.25
[a]1	50	1,000	....	....	....	2.35

SAMPLE NO. 7265. (SALT LAKE CITY, UTAH. 0-12 INS. EFFERVESCED WITH HYDROCHLORIC ACID. TOTAL SALTS, 1.64 PER CENT.)

1	10	1,000	0.074	0.181	0.897	0.177
2	10	1,000	.082	.166	.904	.164
3	10	1,000	.065	.215	.922	.156
1	25	1,000	.06	.10	.716	.200
1	50	1,000	.036	.066	.61	.198
[a]1	50	1,000	.034	.14	.56	.29

[a] Analyses made by Bailey E. Brown in the course of the regular routine of analytical work of the laboratory.

Analyses made by B. E. Brown.

SAMPLE NO. 6604.

Time in days.	Ratio.		CO_3.	HCO_3.	Cl.	SO_4.
	Soil.	Water.				
	Grams.	*cc.*				
1	1	100	0.00	0.24	0.175	1.67
2	1	100	.00	.24	.175	1.67
6½	1	100	.00	.24	.175	1.58

SAMPLE NO. 6676.

1	1	100	8.10	7.75	Trace.	1.68
2	1	100	7.83	8.05	Trace.	1.64
6½	1	100	8.16	7.14	Trace.	1.68

SAMPLE NO. 8083.

1	1	100	0.00	0.48	1.12	0.69
2	1	100	.00	.51	1.12	.68
6½	1	100	.00	.51	1.16	.67

The results are in the main in harmony with the results obtained last year except that the solubility of sulphates does not appear to be as much affected by concentration and time as was indicated by the figures obtained last year. This is doubtless due to the fact that none of the samples used this year contained relatively large amounts of soluble sulphates, the highest percentage being 2.60, while last year samples containing as high as 14.50 per cent were used.

The referee still thinks that the solubility of sulphates is influenced both by time and concentration. Indeed Cameron[a] has shown that under laboratory conditions the maximum solubility of gypsum in sodium chlorid was not reached in much less than 72 hours. In sulphate solutions it would doubtless be slower. As calcium sulphate is a beneficial rather than an injurious compound, and in many cases the time of solution may be indefinitely prolonged, it is necessary to arbitrarily limit it and from 20 to 24 hours appears to be the most suitable period. It is regretted that those chemists who have to deal with the alkali problem did not participate in the work.

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[a] U. S. Dept. of Agr., Bureau of Soils Bul. No. 18.

SOIL ACIDITY.

The referee has devoted a great deal of time in the past three years to a study of soil acidity and methods for its estimation. Some of the statements made are based on work the details of which will be published elsewhere. The work has been confined to the limewater method and the sodium-chlorid method. In the first work done with the sodium-chlorid method it was at once observed that in titrating the solution with alkali a precipitate was always formed. This was usually found to be aluminum hydrate, and careful investigation showed that this precipitate calculated to aluminum oxid required practically all the alkali used in the titration, assuming that a solution of aluminum chlorid was being titrated. The figures obtained are given in Table III.

TABLE III.—*The relation between alkali required and dissolved acid-salt-forming bases.*

Description of sample.	Amount of N/20 alkali required by—			Bases in 100 cc alkali filtrate.		Amount of N/20 alkali required by—		
	1. 100 cc sodium chlorid filtrate.	2. 100 cc water filtrate.	3. 100 cc sodium chlorid sol. acids.	4. Ferric, aluminum, and manganese oxids.	5. Zinc oxid.	6. Column 4, expressed as aluminum oxid.	7. Zinc oxid.	8. Not accounted for by aluminum and zinc oxids.
	cc.	*cc.*	*cc.*	*mg.*	*mg.*	*cc.*	*cc.*	*cc.*
Fine sand, rich in organic matter	3.7	2.7	1.0	1.4	....	1.5	....	2.7
Rhode Island station, very acid	1.6	1.0	.6	2.0	....	2.0	....	0.0
Kaolin, good grade	1.6	.7	.9	1.2	....	1.3	....	.3
Kaolin, good	4.2	Not det.	....	3.0	....	3.3	....	.9
Vegetation experiments, Bureau Chemistry, California soil:								
Limed	5.8	1.0	4.3	2.2	8.6	2.4	3.6	0.0
Not limed	19.0	3.4	15.6	2.6	29.4	3.0	12.2	3.8
Vegetation experiments, Bureau Chemistry, Illinois station	2.2	Alkaline.	2.2	1.0	....	1.1	....	1.1
Carlyle silt loam, subsoil, Illinois	31.5	Not det.	....	30.4	....	33.4	....	0.0
Plot No. 6, fertilizer experiments, Pennsylvania station	.6	Alkaline.	....	.6	....	.6	....	0.0
Plot No. 10, phosphoric acid experiments, Maryland station	1.2	....	....	.5	....	.6	....	.6
Sand from propagating house.	25.7	24.0	1.7	28, Fe_2O_3	....	21.5	....	4.2
Collington loam, glauconitic, Prince George County, Md.	22.6	....	....	22.8	....	23.5	....	0.0

TABLE III.—*The relation between alkali required and dissolved acid-salt-forming bases*—Continued.

Description of sample.	Amount of N/20 alkali required by—			Bases in 100 cc alkali filtrate.		Amount of N/20 alkali required by—		
	1. 100 cc sodium chlorid filtrate.	2. 100 cc water filtrate.	3. 100 cc sodium chlorid sol. acids.	4. Ferric, aluminum, and manganese oxids.	5. Zinc oxid.	6. Column 4, expressed as aluminum oxid.	7. Zinc oxid.	8. Not accounted for by aluminum and zinc oxids.
	cc.	*cc.*	*cc.*	*mg.*	*mg.*	*cc.*	*cc.*	*cc.*
Loam, District of Columbia, very acid, growth only sorrel	22.8	....	....	21.8	....	23.0	....	0.0
Clay subsoil, Piedmont region.	2.8	....	....	1.8	....	2.0	....	.8
Fine sand, rich in organic matter, treated with—								
Normal sodium chlorid	3.6	.7	2.9	2.3	....	2.5	....	1.1
Double normal sodium chlorid	5.2	.7	4.5	3.5	....	3.9	....	1.3
Normal potassium chlorid.	6.6	.7	5.9	5.0	....	5.0	....	1.1
Double normal potassium chlorid	8.0	.7	7.3	6.9	....	7.6	....	.4
Brick clay treated with—								
Normal sodium chlorid	22.0	.4	21.6	17.8	....	19.1	....	2.9
Double normal sodium chlorid	25.3	.4	24.9	20.0	....	22.0	....	3.3
Normal potassium chlorid.	27.6	.4	27.2	20.6	....	22.2	....	5.4
Double normal potassium chlorid	29.6	.4	29.2	21.6	....	23.2	....	6.4

These results show conclusively that the apparent acidity of the solution is due to the presence of an acid salt, usually aluminum chlorid. In no instance was the referee able to find an appreciable amount of free hydrochloric acid, even in the most acid of these solutions. The test used was that previously given and its applicability here can be easily proved. Solutions of other salts and other strengths of sodium chlorid have also been tried, and the results of these experiments are given in Table IV.

TABLE IV.—*Sodium-chlorid method; acidity by different solvents of varying strength.*

Description of sample.	Soil.	Kind and strength of solvent.	N/20 alkali to 100 cc of filtrate.	Acidity, parts per million.
	Grams.		*cc.*	
Brick clay (moist sample)	200	5 per cent sodium chlorid	28.0 29.0	980 1,015
Do	200	10 per cent sodium chlorid	40.0	1,400
Do	200	20 per cent sodium chlorid	40.0	1,400
Do	100	5 per cent sodium chlorid	19.2 19.0	1,344 1,300
Do	100	10 per cent sodium chlorid	25.0 26.0	1,750 1,820
Do	100	20 per cent sodium chlorid	25.5 26.5	1,785 1,855
Do	100	5 per cent sodium sulphate	18.8	1,316
Do	100	5 per cent potassium sulphate	22.4 23.0	1,568 1,610

Table IV.—*Sodium-chlorid method; acidity by different solvents of varying strength*—Continued.

Description of sample.	Soil.	Kind and strength of solvent.	N/20 alkali to 100 cc of filtrate.	Acidity, parts per million.
			cc.	
Brick clay (air-dry sample)	100	Normal sodium chlorid	22.0 23.0	1,540 1,610
Do	100	2 normal sodium chlorid	25.3	1,771
Do	25	Normal sodium chlorid	7.5	2,100
Do	50	Normal sodium chlorid	13.5	1,890
Do	100	Normal potassium chlorid	27.6	1,932
Do	100	2 normal potassium chlorid	29.6	2,072
Sandy soil, rich in organic matter	100	Normal sodium chlorid	3.6	252
Do	100	2 normal sodium chlorid	5.2	364
Do	25	Normal sodium chlorid	1.7	476
Do	50	Normal sodium chlorid	2.5	350
Do	100	Normal potassium chlorid	6.6	462
Do	100	2 normal potassium chlorid	8.0	560

The acidity appears to vary with the kind and strength of the salt solution, but the variation can not be foretold nor is the assumption justified that any particular strength or kind of a salt solution is a measure of the lime requirements of soils. The originators of the method found that the reaction taking place was not complete, and proposed a factor of 1.5 and later of 2 to correct for this condition. It was found that the brick clay reported in Table IV required the factor 1.3, while the sand rich in organic matter required a factor of 2. It is evident, therefore, that the same factor is not applicable to all soils and that a large error is introduced by the use of a single factor. The determination of the factor for every soil would lengthen the method greatly.

It has also been found that a soil containing organic matter, limed in accordance with the results obtained by this method, is seldom neutral but still is acid, according to the method. Some faintly alkaline soils also show acidity by this method, as is seen in Table V.

It is concluded that this method does not show the acidity due to water insoluble organic acids, that there is no quantitative setting free of hydrochloric acid by such organic acids, and that the apparent acidity as thus determined is due to the water soluble acids and acid salts plus the acid salts which are produced by the action of the salt solution upon the hydrous or other silicates of the soil. The method in its present form has nothing like the accuracy claimed for it by the originators. If the acidity requires 20 or more cubic centimeters of N/20 alkali per 40 grams of soil, duplicates may differ from one-half to 1 cc of alkali, which is equal to 35 to 70 parts per million, and the multiplication of this error by an erroneous factor may increase the actual error to as much as 600 parts per million.

Several modifications of the limewater method have been tried, the results of which are given in Table V. No modification presented any advantage over the original method and the table calls for no detailed discussion:

TABLE V.—*Acidity expressed as parts of calcium oxid per million of soil.*

Description of sample.	Sodium chlorid method.	Calcium hydroxid method.			
		Dried on bath.		Dried in air; stood 16 hours.	Not dried; stood 16 hours.
		Stood 16 hours.	Stood 40 hours.		
Sandy soil rich in organic matter, Norfolk, Va	258	4,000	3,400	3,900	3,500
Sandy soil rich in organic matter, after cropping	252	3,900	3,200	>3,400	2,100
Sandy soil rich in organic matter, limed sodium chlorid method 200 parts per M	161	3,800	2,700	....	....
Sandy soil rich in organic matter, limed 1,700 parts per M	49	2,400	1,500	<1,900	800
Loam, very acid, growth sorrel	1,365	....	2,200	....	2,500
Loam, very acid, growth sorrel, after cropping	1,631	2,400	2,000	<2,000	2,000
Loam, very acid, growth sorrel, after cropping, limed 1,200 parts	658	2,100	1,200	....	1,600
Loam, very acid, growth sorrel, after cropping, limed 2,250 parts per M	91	1,200	600	<1,000	600
Pot No. 67, P_2O_5 experiments, calcium oxid series, Rhode Island station	112	1,000	600	> 800	800
Pot No. 67, vegetation experiments, Bureau of Chemistry, California soil, limed 1,200 parts	370	900	....	....	....
Pot No. 68, vegetation experiments, Bureau of Chemistry, California soil, unlimed	1,330	2,000	2,000	1,900	1,600
Pot No. 35, vegetation experiments, Bureau of Chemistry, Illinois station	153	(a)	....	....	....
Kaolin No. 1	112	100	100	100	100
Kaolin No. 2	280	150	....	....	....
Carlyle silt loam, Illinois	2,170	2,600	<2,400	2,600	2,400
Coarse sand from propagating house	1,800	1,000	....	....	....
Collington loam, subsoil, glauconitic, Maryland	1,470	1,100	....	....	....
Brick clay, 4 feet below surface, Maryland	1,610	1,400	....	<1,300	1,200
Calcium oxid plot, 10 bushels per acre, 0 to 6 inches, Maryland station	196	500	....	< 400	300
Calcium oxid plot, 20 bushels per acre, 0 to 6 inches, Maryland station	63	300	....	....	200
Calcium oxid plot, 30 bushels per acre, 0 to 6 inches, Maryland station	28	(a)	....	....	....
Calcium oxid plot, 0 bushels per acre, 0 to 6 inches, Maryland station	406	600	....	< 500	400
Plot No. 4, "soil test" plot, Rhode Island station, taken 1894	518	1,800	....	<1,700	1,700

[a] Alkaline.

The fact that long standing of the treated and dried soil in contact with water shows less acidity than short standing is interesting and seems to indicate the breaking up of the more insoluble calcium silicates on long standing. The method is rather long and cumbersome and for this reason is objectionable. It is accurate to within 300 to 400 parts per million under extreme conditions, i. e., where the supernatant liquid is highly colored by organic matter and where the acidity is high. On soils of low acidity which give colorless, clear liquids the results are accurate to within 100 parts per million. Both methods are simple in their details and it does not appear worth while to devote further analytical study to them until a more definite knowledge is possessed as to the actual lime requirements of soils as a basis for such investigations.

RECOMMENDATIONS.

It is recommended—

(1) That in the continuation of the work on methods for available phosphoric acid the referee compare greater concentrations than N/200 acid.

(2) That the work on the solubility of phosphoric acid and potash in the different soil layers be continued.

(3) That the referee investigate methods for determining total phosphoric acid.

(4) That the following provisional method for the analysis of the water soluble portion of alkali soils be incorporated in the methods:

PROPOSED PROVISIONAL METHOD FOR THE DETERMINATION OF WATER SOLUBLE CONSTITUENTS OF ALKALI SOILS.

(a) *Preparation of solution.*

Place 25 grams of soil in a graduated 500 cc flask, fill to the mark with distilled water, allow to stand twenty to twenty-four hours, shaking frequently, and filter through a dry filter, or if clear, siphon off the supernatant liquid.

(b) *Determination of total carbonates.*

Titrate an aliquot portion of this filtrate with N/20 sulphuric acid, using a few drops only of methylorange as indicator, and be very careful not to run over the end point. From the number of cubic centimeters of acid required calculate the total CO_2 as carbonates.

(c) *Determination of chlorin.*

To the aliquot portion used for the titration of carbonates add a few drops more of N/20 acid, then a little potassium chromate, and titrate with N/10 silver nitrate and calculate to chlorin (cc N/10 silver nitrate multiplied by 0.003518=the number of grams of chlorin).

(d) *Determination of sulphuric acid.*

An aliquot portion of the original filtrate is made slightly acid with hydrochloric acid, and if clay is flocculated the solution is filtered and the filter washed free of sulphates. To the boiling solution add slowly a large excess of barium chlorid solution and finish the determination as in 4 (j)[a]. In solutions containing large amounts of carbonates the ignited barium sulphate should be treated with hydrofluoric acid and a few drops of sulphuric acid ignited and again weighed.

(e) *Determination of bases.*

If the original filtrate is turbid, as it is apt to be when the soil contains carbonates and but little of other salts, it may be cleared by adding 5 cc of thick alumina cream to 200 or 300 cc and filtering through a dry filter. In using portions of this filtrate for the determinations, correction should be made for the added alumina cream. To aliquot portions of the solution add hydrochloric acid in excess, and in cases where silica may be present, separate in the usual way and determine the bases as in 4 (b), (c), (d), and (e).[b]

(f) *Determination of phosphoric acid.*

Add an excess of nitric acid to an aliquot portion of the original solution. Make nearly neutral with ammonia, precipitate with ammonium molybdate, finishing the determination as in 4 (g).[c]

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[a] U. S. Dept. of Agr., Bureau of Chemistry Bul. No. 46, Methods of Analysis, p. 75.

[b] Ibid., pp. 72, 73.

[c] Ibid., p. 74.

(5) That on page 72, under 4 (a), "Acid digestion of the soil," the tenth line be changed to read, "filter, wash free of chlorids, and again evaporate the filtrate to complete dryness as before."

(6) That on page 74, under 4 (g), determination of phosphoric acid, the official method given be marked "(a)" and the following optional provisional method be inserted as (b):

(b) *Optional provisional method:* Proceed as in (a) "until all the phosphoric acid is precipitated" and then finish the determination as follows:

After standing for three hours at a temperature not above 50°, filter on a small filter, wash with water until two fillings of the filter do not greatly diminish the color produced with phenolphthalein by one drop of standard alkali. Place the filter and precipitate in the beaker and dissolve in standard alkali, add a few drops of phenolphthalein solution, and titrate with standard acid, 1 cc of which equals 0.0005 gram of phosphoric acid (P_2O_5).

(7) That on page 74, 4 (h), the word "available" be omitted from the heading "Provisional method for the determination of available phosphoric acid."

(8) Under 4 (k), "Determination of potash and soda," insert in sixth line, after "hot water," Finish by (1) or (2).

(1) Add ammonia, etc. (present method).

(2) To the hot filtrate add slowly a few cubic centimeters of ammonium sulphate (75 grams salt per liter) and after the precipitate has settled filter and wash thoroughly with hot water, evaporate the filtrate to dryness, and ignite. Add a little hot water, and if any material is not dissolved filter, wash, evaporate to dryness and ignite as before. Add a little ammonium carbonate, volatilize to break up bisulphates. Heat to redness and weigh as sulphates.

(9) That on page 76, paragraph 10, "Determination of humus," eleventh line, insert after the word "filtered" "the filtrate must be perfectly clear and free from turbidity."

(10) That the retiring referee be instructed to make such verbal corrections as these changes will involve and submit them to the secretary for approval and publication.

Mr. Lipman. I have a paper on soils to submit.

THE FIXATION OF ATMOSPHERIC NITROGEN BY BACTERIA.

By J. G. Lipman.

Important discoveries in the domain of agricultural bacteriology have often been preceded by repeated failure. No better example need be cited than the history of that well-known problem, the fixation of atmospheric nitrogen by the joint activity of *Bacillus radicicola* and of leguminous plants. One need but recall the work of Boussingault,[a] and the work of Lawes, Gilbert, and Pugh,[b] and their painstaking care and the conscientious attention to detail, to realize the limitations of even the best of investigators. Their failure is no less instructive than the partial success of Atwater[c] and the complete success of Hellriegel and Wilfarth.[d] Both the failure and the success lay a strong emphasis on the importance of the biological properties of arable soils as a factor in the economy of plant growth. It is a significant fact, and not devoid of encouragement to those who are interested in the progress of agriculture, that the soil chemist and the soil physicist have come to a better appreciation of their common aims and have learned to work together. And it is to be hoped that in the study of soil problems the proper place will be accorded to soil bacteriology. Nothing but good can result from this recognition. It will be found that soil bacteriological studies will help to explain many a seemingly inexplicable result obtained in field and vegetation experiments.

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[a] Compt. rend., 1859, 48: 303; Agronomic, etc., 1860, 2d ed., vol. 1.

[b] Philosophical Transactions, 1861, part II; Philosophical Transactions, 1888.

[c] Amer. Chem. J., vol. 6, No. 6; vol. 8, No. 5.

[d] Beilag. Zeit. Ver. Rübenzucker-Ind. d. d. R., 1888.

The fixation of atmospheric nitrogen by soil bacteria in cooperation with higher plants has been the subject of much thought and of much study, and we all know what the achievements in this field of research have been; but as to the fixation of elementary nitrogen by bacteria independently of higher plants very little is known. The collection of facts bearing on this point is of recent origin, and most of these facts have been brought to light within the last two years. A rapid survey of the subject and its historical development will show its present status.

In his second series of experiments on the fixation of atmospheric nitrogen Boussingault obtained results which showed a gain of nitrogen in the soils experimented with. He grew leguminous plants in a mixture of rich garden soil and sand, and found at the end of the experiment a gain of 71.4 mg of nitrogen on the 359.7 mg supplied in the seed and soil. That was in 1858. In the following year he repeated the experiment, and obtained a gain of 67.1 mg on a total of 338.0 mg of nitrogen supplied in the soil and in the seed. Stranger still, he noted that there was more gain in the soil itself than in the plants; that the soil, moreover, gained not only in nitrates and ammonia, but organic matter also, possibly the remains of living organisms. To use his words as transcribed by Gilbert:

> Vegetable earth contains not only dead organic matter, but living organisms—germs—the vitality of which is suspended by drying and reestablished under favorable conditions as to moisture and temperature. This mycodermic vegetation is not always visible to the naked eye and its progress must be followed by the aid of the microscope. The mycoderms have only an ephemeral existence, and they leave their detritus in the soil, which in time may give rise to ammonia and to nitric acid. Even if the nitrogen of the air takes part in nitrification, a part of the nitrogen will exist in mycoderms and their remains.

Evidently Boussingault had in his hand facts which might have revealed to him the cause of these gains had contemporary information been sufficient to throw greater light on the subject. Hellriegel and Wilfarth's discovery of symbiotic fixation was still almost thirty years away, Berthelot's study of nitrogen-fixing bacteria, Winogradsky's isolation of nitrifying bacteria and of the nitrogen-fixing *Clostridium pasteurianum* were not to come for more than three decades, and in fact the technique of modern bacteriology was practically unknown. Small wonder, then, that, clear sighted as he was, Boussingault failed to recognize the significance of his analytical results. He deliberately eliminated them from his later studies, and placing more reliance on his later experiments (1860–1871), he wrote to Gilbert in 1876:

> Quant a l'absorption de l'azote gazeux de l'air par la terre végétale je ne connais pas une seule observation irréprochable qui l'etablisse; non seulement la terre n'absorbe pas d'azote gazeux mais elle en émet, ainsi que vous l'avez reconnu avec Mr. Lawes, comme l'a vu Reiset pour le fumier, comme nous l'avons constaté, M. Schloesing et moi, dans nos recherches sur la nitrification. S'il est en physiologie un fait parfaitement démontré, c'est celui de la non assimilation de l'azote libre par les végétaux et je puis ajouter par les plantes d'un ordre inferieur, telles que les mycodermes, les champignons.

Gilbert comments on this as follows:

> It is remarkable that in that letter he should so expressly give his opinion against the supposition that the lower organisms within the soil affect the fixation of free nitrogen, notwithstanding the evidence of his experiments of 1858 and 1859 that the gain, if there really were gain, was chiefly by the soil, and chiefly as organic matter, the accumulation of which he attributed to the development of mycodermic vegetation. It is true that in the discussion of the results he did not give any clear indication whether he considered that the apparent fixation was due in the first instance to the process of nitrification, the mycoderms only appropriating the nitrogen of the

nitrates formed, or whether he supposed that the mycoderms themselves were the primary agents, and that the nitrification was only the result of the oxidation of the mycodermic remains.

It did, indeed, seem that in the results in question there was *the germ of the germ explanation* of the fixation of free nitrogen, if such took place at all, in connection with vegetation. But we confess that Boussingault's very distinct conclusion against the assumption of any such agency, notwithstanding the indications of some of his own experiments, leads us still to ask further confirmation of the evidence of others in the same direction, which has been accumulating during the last few years.

Gilbert made the above statement in 1888, thirty years after Boussingault had made the observation in question. He, too, was still inclined to doubt that there might be fixation of nitrogen in arable soils, notwithstanding the fact that Berthelot[a] had already a considerable amount of analytical data showing that under certain conditions bare soils can increase their content of combined nitrogen at the expense of the free nitrogen of the air. Working with argillaceous sand, with clay, and with crude kaolin, he obtained[b] in six months (April to October, 1885) an increase in every case, the greatest gain being made by the argillaceous sand. When the soil was placed in a large 4-liter flask and sterilized no gain was observed, while a soil similarly treated but not sterilized showed a gain of nitrogen. At certain intervals samples of the soil were taken and analyzed, the analyses showing a gradual and steady increase in the total combined nitrogen, while the amount of nitrate produced was small and the slight amount of ammonia originally present either remained constant or tended to decrease. The results thus obtained indicate that the nitrogen gained by the soil was in the insoluble organic form, and this, taken together with the fact that the gains were made in unsterilized vessels and largely during the warmer part of the year, justifies the assumption that the fixation was accomplished by living organisms. Without going into further detail, it is sufficient to state here that corroborative evidence as to the fixation of nitrogen by soils and soil organisms was also furnished by Joulie,[c] Dietzell,[d] Tacke,[e] Gautier and Drouin,[f] Schloesing and Laurent,[g] Frank,[h] Koch and Kossowitch,[i] Kossowitch,[j] Krüger and Schneidewind,[k] Richter,[l] Petermann,[m] Immendorff,[n] Deherain,[o] Pagnoul,[p] Pickard,[q] Breal,[r] and Kühn.[s] Not content with proving that there may be an accumulation of nitrogen in bare and uncropped soils, Berthelot[t] also attempted to isolate the specific organisms to which such fixation is due. With the aid of Professor Guignard he made inoculations into sterile bouillon from a garden soil, and after incubation at 20° and proper dilution with sterile bouillon gelatin plates were prepared. Inoculations were made from the colonies which appeared on the plates, and the bacteria thus isolated were tested for their nitrogen-fixing power. A careful description of these organisms is lacking, so that their identification is impossible. Berthelot gives, however, some general statements concerning them, and, what is more important, shows by his analytical results that there was a gain of combined nitrogen in some cases. He concludes from these experiments that there are chlorophylless bacteria in the soil capable of fixing elementary atmospheric nitrogen. They require organic carbon and hydrogen and enough combined nitrogen to promote their initial growth. When the amount of combined nitrogen present becomes considerable, the bacteria prefer to cover their nitrogen requirements by utilizing the combined soil nitrogen, and for this reason the fixation of soil nitrogen has its limits. It is quite evident from the above that Berthelot had cleared the ground for more exact bacteriological studies. To him belongs the credit of having proved conclusively that the fixation of gaseous atmospheric nitrogen may be accomplished in arable soils by virtue of certain micro-organisms contained in them. His skill as a chemist enabled him to overcome the many difficulties involved in the analytical work and to render his researches of permanent interest.

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[a] Chimie Vegetale et Agricole, Paris, 1899.

[b] Compt. rend., **101**: 775; Idem, **104**: 209, 630.

[c] Bul. de la Soc. des Agric. de France, 1886, No. 1, pp. 19–29.

[d] Naturforscher. Versaml., Magdeburg, 1884.

[e] Landw. Jahrb., 1889, **18**: 439.

[f] Compt. rend., 1888, **106**: 570; Idem, 1892, **115**: 572.

[g] Compt. rend., 1888, **106**: 1607.

[h] Landw. Jahrb., 1888, **17**: 421; 1890, **19**: 588.

[i] Bot. Ztg., 1893, **51**: 321.

[j] Bot. Ztg., 1894, **52**: 97.

[k] Landw. Jahrb., 1900, p. 771.

[l] Landw. Versuchs-Stat., 1899, **51**: 221.

[m] Abstr. Chem. Centr., 1893, **1**: 988.

[n] Landw. Jahrb., 1892, **21**: 281.

[o] Compt. rend., 1897, **125**: 278.

[p] Ann. agron., 1890, **16**: 250; Abstr. Ex. Sta. Rec., **3**: 120.

[q] Compt. rend., 1892, **114**: 81.

[r] Ann. agron., 1892, **18**: 269; Abstr. Ex. Sta. Rec., **4**: 375.

[s] Abstr. Centr. f. Bact., pt. 2, **7**: 601.

[t] Compt. rend., 1893, **116**: 842.

At this point the problem was attacked by Winogradsky,[a] whose admirable mental equipment made it possible for him to shed much light on the subject. His first communication dates back to 1893 and deals with experiments that were carried out with impure cultures of a butyric ferment. The nutritive solutions employed by him were carefully freed from the last traces of nitrogen, and contained besides the mineral salts a fermentable organic compound, dextrose. Butyric fermentation was produced in these solutions by a long spore-bearing bacillus, which developed normally in the absence of combined nitrogen. Winogradsky had not succeeded at this time in isolating this bacillus in pure culture. In the above experiments it was accomplished by two other bacilli. It seemed, however, that the latter took no part in the fixation of free nitrogen, for they refused to grow in media free from nitrogen, but grew vigorously in solutions to which small quantities of ammonium salts were added. This, taken together with the fact that they produced neither gas nor butyric acid, led Winogradsky to believe that these accompanying bacteria were incapable of fixing free nitrogen, although they were capable of growing in solutions very poor in combined nitrogen. At the end of the experiment the solutions were evaporated to dryness under diminished pressure and analyzed according to the Kjeldahl method. It was found that the maximum fixation was attained where no combined nitrogen was purposely added, namely, with 3.1 mg of nitrogen for every gram of dextrose used. When combined nitrogen was added to the culture solution, the fixation of nitrogen was diminished. Thus, with 2 grams of dextrose and 1.8 mg of combined nitrogen in the solution there were 1.7 mg of nitrogen fixed, while with 4 grams of combined nitrogen added the fixation was only 0.6 mg of nitrogen.

In a later communication Winogradsky[b] reports experiments with pure cultures of the butyric ferment. He isolated them by anaerobic culture methods, and found that aerobic growth takes place only in the presence of certain aerobic bacteria, which serve to diminish the oxygen pressure. With the two accompanying bacteria mentioned elsewhere and the butyric ferment he noted a diminution in the amount of nitrogen fixed as the concentration of the dextrose solution or the amount of initial nitrogen increased. When the amount of combined nitrogen added reached 21.2 mg for every gram of dextrose, there was even a loss of nitrogen (2.2 mg). Winogradsky concludes from these experiments that the gain of nitrogen depends on the proportion of the combined nitrogen to the sugar. In order that any gain may be obtained the proportion of combined nitrogen to the dextrose should be 6:1,000. Because of the characteristic formation of *Clostridia*, Winogradsky named this bacillus *Clostridium pasteurianum*.

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[a] Compt. rend., 1893, **116**: 1385.

[b] Compt. rend., 1894, **118**: 353.

The researches of Berthelot and Winogradsky soon led to attempts at the practical development of the subject, and this received a decided impetus when Caron, of Ellenbach, Germany, made public the results of his investigations.[a] These soon attained considerable notoriety and led to the commercial exploitation of his culture. The firm of Friedrich Bayer & Co., of Elberfeld, Germany, placed on the market a bacterial preparation under the name of Alinit. This preparation, the manufacturers claimed, would enable nonleguminous crops to make use of free atmospheric nitrogen provided that certain precautions be observed.

These claims led to an extended study of Alinit by men like Stoklasa,[b] Lauk,[c] Stutzer and Hartleb,[d] Kolkwitz,[e] Gain,[f] Lutoslawski,[g] Schultze,[h] Kruger and Schneidewind,[i] and Sewerin.[j] A careful study of the evidence submitted by these investigators shows that *B. ellenbachensis* of the Alinit preparation is incapable of fixing atmospheric nitrogen. Attempts were also made by Frank,[k] Immendorff,[l] Gonnermann,[m] and others to determine whether *B. radicicola* is capable of fixing atmospheric nitrogen, when developing outside of the legume tubercles, but with doubtful[n] or negative results, while Maze,[o] on the other hand, claims that he succeeded in obtaining positive results.

Then came Beijerinck's communication in 1901,[p] dealing with a new group of bacteria. Space will hardly permit a lengthy review of this statement and the subsequent report by him. Suffice it to say that he gave the name of Azotobacter to the entire group, and described two members *A. chroococcum* and *A. agilis*. The former was found to occur in most cultivated soils, the latter in the canal water at Delft. These two, together with *A. vinelandii* isolated by the writer from a Vineland soil in south Jersey, constitute, in so far as I am aware, all the known members of this interesting group of bacteria. Beijerinck, together with Van Delden,[q] found that *A. chroococcum* can fix only very slight quantities of nitrogen, if any at all, when growing in pure culture, and that growth stops before the carbon source is exhausted. Large amounts of nitrogen are fixed only when it is growing in symbiosis with other organisms. Gerlach and Vogel,[r] on the other hand, isolated an organism which they found capable of fixing considerable amounts of atmospheric nitrogen when growing in pure culture. They believe that the organism isolated by them is identical with Beijerinck's *A. chroococcum*.

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[a] Landw. Versuchs-Stat., 1895, **45**: 401.

[b] Centr. f. Bact., etc., 1898, pt. 2, **4**: 39, 78, 119, 284, 507, 535.

[c] Idem, p. 290.

[d] Idem, pp. 31, 73; Idem, 1899, pt. 2, **5**: 706; Bot. Centr., **72**: 229.

[e] Centr. f. Bact., etc., pt. 2, 1899, **5**: 670.

[f] Rev. gen. de botaniq., 1899, **11**: 18–28; Centr. f. Bact., etc., 1899, pt. 2, **5**: 847.

[g] Deut. Landw. Presse, 1898, **25**: 920.

[h] Landw. Jahrb., 1901, **30**: 319.

[i] Landw. Jahrb., 1899, **28**: 579.

[j] Centr. f. Bact., etc., 1902, pt. 2, **9**: 712, 746.

[k] Bot. Ztg., 1893, **51**: 139.

[l] Landw. Jahrb., 1892, **21**: 218.

[m] Landw. Jahrb., 1894; Jacobitz, Centr. f. Bact., etc., pt. 2, **7**: 783, 883, 876.

[n] Beijerinck, Bot. Zt., **46**; Jacobitz, Centr. f. Bact., etc., pt. 2, **7**: 783, 883, 876.

[o] Annal. Inst. Pasteur, 1898, **12**: 1, 128; Abst. Ex. Sta. Rec., 1898, **10**: 318.

[p] Centr. f. Bact., etc., pt. 2, **7**: 561.

[q] Idem, 1902, pt. 2, **9**: 3.

[illegible], pt. 2, **8**: 669; 1903, pt. 2, **10**: 636.

The researches of Beijerinck and Van Delden, of Gerlach and Vogel, and of Winogradsky,[a] were carried out with specific bacteria which have been described sufficiently to allow their recognition by others. Their work shows that there are at least two classes of bacteria capable of fixing large quantities of nitrogen, namely, spore-forming anaerobic and nonspore-forming aerobic organisms. Practical attempts to decide whether they can be used to increase the fertility of the soil are wanting if we except the investigations of Gerlach and Vogel, and of Freudenreich.[b] But from the theoretical standpoint we have to deal here with a group of very interesting bacteria. In some still unexplained way they can break up the molecules of elementary nitrogen and cause it to combine with other elements to form ultimately the albuminoids of the bacterial body. It is not known whether the albuminoid molecules are the first product of the synthesis or not, but it is believed that simpler nitrogenous substances are first formed, although these have thus far escaped detection. Certain it is that the solution of this problem will help us to understand the synthetical processes, not only of the bacterial cell, but all the synthetical processes in the cells of higher plants.

The Isolation of Nitrogen-Fixing Bacteria of the Azotobacter Group.

The solutions employed for the purposes of isolation had the following composition:

Tap water	cubic centimeters..	1,000.00
Mannite	grams..	20.00
Potassium phosphate (K_2HPO_4)	gram..	.5
Magnesium sulphate	do....	.2
Calcium chlorid	do....	.02
Potassium nitrate	do....	.005

The whole was made neutral to phenolphthalein with sodium hydrate, and after sterilization in the autoclave at 1.5 atmospheres pressure was inoculated with a small quantity of fresh soil. The crude culture thus obtained by inoculation with a Vineland soil was reinoculated into a similar culture containing 20 grams of glycerin in place of the mannite. In ten days reinoculations were made into fresh glycerin solutions, and from these stroke cultures were made on mannite agar. In 3 days at 28° there appeared on these plates, among others, whitish, dense, raised colonies. Microscopical examination showed them to consist of azotobacter bacteria, and inoculation was accordingly made into glucose solutions, composed like the others, but with glucose instead of mannite or glycerin. Evidence of vigorous growth became apparent in 3 or 4 days, and the microscopical examination showed an apparently pure culture of organism occurring as short rods or large cocci, with many of the former actively motile. These were replated three times on mannite agar, and in each case macroscopical as well as microscopical examination showed only one kind of colonies and one kind of organisms. Inoculation into meat extract bouillon left the latter clear, although a very slight precipitate was formed here. Microscopical examination also here showed the presence of the same organism. The cultural characteristics in various media showed it to be essentially different from the organisms described by Beijerinck, and it was therefore named *A. vinelandii*.

In order to show its behavior in nitrogen-free or nitrogen-poor solutions, the experiments following are presented.

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[a] Centr. f. Bact., etc., 1902, pt. 2, 9: 43.

[b] Idem, pt. 2, 10: 514.

SERIES VI.—*100 cc glucose solution.*

No. 1, size of flask, 250 cc, sterile.
No. 2, size of flask, 250 cc, inoculated.
No. 3, size of flask, 500 cc, inoculated.
No. 4, size of flask, 1,000 cc, inoculated.

After 10 days the cultures were analyzed with the following results:

No.	Ammonium hydrate (0.003038 gram nitrogen per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.20	0.61	....
2	.75	2.28	1.67
3	1.25	3.80	3.19
4	2.80	8.51	7.90

These tabulations show that the fixation of nitrogen by *A. vinelandii* increased with the surface exposure. This circumstance is clearly due to two reasons. In the first place the bacteria of the azotobacter group are aerobic organisms, and with them the fixation of nitrogen takes place where the access of oxygen is greatest; that is, at the surface. Where the surface exposure is greatest there also growth is greatest, and where growth is more vigorous the building of the organic nitrogenous substance in the bacterial bodies is also most active. In the second place, the larger surface exposed places at the disposal of the bacteria not only a greater amount of oxygen but also a greater amount of gaseous nitrogen, and this is utilized readily when the bacterial growth is vigorous.

SERIES VIII.

In growing *A. vinelandii* in mannite solutions in which strips of filter paper had been placed, it was noticed that in the presence of the latter growth was far more abundant than in the tubes where no paper was present. The question naturally arose whether *A. vinelandii* is capable of utilizing part of the filter paper as a source of carbon and whether by its aid it is capable of fixing greater quantities of nitrogen. To decide this point the following experiment was arranged:

1. 150 cc mannite solution + 1 gram calcium carbonate.
2. 150 cc mannite solution + 2 grams filter paper.
3. 150 cc mannite solution.
4. 150 cc mannite solution + 2 grams filter paper.

The mannite solution used contained 5 mg of potassium nitrate per liter and the flasks were the ordinary Jena, round-bottomed flasks, used for the Kjeldahl digestion. At the end of the experiment it was merely necessary to take out the cotton stopper, add mercury and sulphuric acid, and proceed with the work as in other Kjeldahl determinations. In this way it became unnecessary to transfer the culture solutions at the end of the experiment, and thus a possible error was avoided. After sterilization in the autoclave and cooling, 3 and 4 were inoculated from a pure culture of *A. vinelandii*, while 1 and 2 were allowed to remain sterile. In 48 hours the several flasks appeared as follows:

1. Clear.
2. Clear.
3. Cloudy, floating membrane.
4. Turbid, floating membrane.

In three weeks the solutions in this series were analyzed and the following amounts of nitrogen were found:

No.	Ammonium hydrate (0.003012 gram nitrogen per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.15	0.45	....
2	.65	1.96	....
3	4.10	12.85	11.90
4	9.85	29.67	27.71

It will be seen from the above that the sterile filter paper contained 1.96 mg less 0.45 mg, or 1.51 mg of nitrogen. Comparing the amounts of nitrogen fixed in 3 and 4, we find that in the presence of the filter paper there was fixed in 4 more than twice as much nitrogen as was fixed in 3. The 150 cc of the mannite solution contained 3 grams of mannite, hence there were fixed in 3, 3.97 mg of nitrogen for every gram of mannite consumed. Disregarding for the moment the filter paper as a source of organic carbon, we find that there were fixed in 4, 27.71 mg of nitrogen for the 3 grams of mannite consumed, or nearly 9.24 mg of nitrogen for every gram of mannite used. It is pertinent to ask here whether part of the filter paper was hydrolyzed and then used by the bacteria as a source of food. Another series of experiments reported below seems to indicate that the filter paper is not used by the bacteria as a source of carbon. It does not follow, however, that such utilization of the cellulose in the soil humus does not take place.

SERIES VIII A.

The arrangement of this series was as follows:

1 and 2. 200 cc mannite solution+1 gram filter paper, sterile.
3 and 4. 200 cc mannite solution+1 gram filter paper, *A. vinelandii.*

At the end of four weeks the cultures were sterilized, the filter paper was removed after careful rinsing, and the residues analyzed. The following amounts of nitrogen were found:

No.	Ammonium hydrate (0.001307 gram nitrogen per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.25	0.32	....
2	.20	.26	....
3	9.00	11.76	11.44
4	8.75	11.44	11.12

The filter paper when dried and weighed showed no appreciable change in weight. Unintentionally the parallel cultures containing no filter paper were left out, so that the results here can not be claimed to prove with certainty that the filter paper is not attacked by a vigorously growing culture of *A. vinelandii;* nevertheless, it would appear from this that where the presence of the filter paper encourages growth it is not necessarily due to the combined carbon offered by the paper to the bacteria.

Series IX.

In this series, as in the preceding, filter paper was used together with mannite. The different proportions of the paper used were intended to show whether there is a point beyond which the increase in the quantity of paper produces no corresponding increase in the amount of nitrogen fixed. The arrangement of the experiment was as follows:

(1) 200 cc mannite solution.
(2) 200 cc mannite solution +0.5 gram paper. } Sterile.
(3) 200 cc mannite solution +1.0 gram paper.
(4) 200 cc mannite solution.
(5) 200 cc mannite solution +0.5 gram paper.
(6) 200 cc mannite solution +1.0 gram paper.
(7) 200 cc mannite solution +2.0 gram paper.

There was also added to each flask 1 gram of calcium carbonate. In four days the several solutions appeared as follows:

(1) Clear.
(2) Clear.
(3) Clear.
(4) Turbid, pigment, surface film.
(5) Turbid, pigment, surface film.
(6) Turbid, pigment, surface film.
(7) Turbid, pigment, surface film.

At the end of 10 days the contents of the flasks were analyzed and the following amounts of nitrogen found:

No.	Ammonium hydrate (0.00302 gram N. per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.15	0.45	....
2	.30	.91	....
3	.40	1.21	....
4	2.30	6.95	6.50
5	3.60	10.87	9.96
6	3.80	11.48	10.27
7	3.75	11.32	9.81

The mannite solution used here was like that used in the other series, except that it contained 0.15 gram of mannite per liter, instead of 0.20 gram; the 200 cc of mannite solution used here contained in each case 3 grams of mannite. With these 3 grams of organic substance there were fixed in 4, 6.40 mg of nitrogen, or 2.17 mg of nitrogen for every gram of mannite used up. In 5, where there was 0.5 gram of filter paper added to the 250 cc of solution, there was a fixation of 9.96 mg, or of 3.32 mg for every gram of mannite used up. When the quantity of paper was still further increased in 6, the amount of nitrogen fixed also increased, and we find here a fixation of 10.27 mg, or of 3.42 mg of nitrogen per gram of mannite employed. A still further increase in the quantity of filter paper used not only failed to yield an increased amount of fixed nitrogen, but actually led to a diminution in the amount of nitrogen fixed. Thus we find in 7 a total fixation of 9.81 mg of nitrogen, or of 3.27 mg per gram of sugar consumed. This is slightly less than the amount fixed in 5, where only 0.5 gram of filter was used. This experiment confirms the results obtained in Series VII, but it also shows that there is a maximum quantity of filter paper that can be advantageously used. Evidently the presence, in 200 cc of the mannite solution, of 2 grams of filter paper was less favorable for the fixation of a maximum amount of nitrogen than the presence in the same solution of only 1 gram of paper. Larger proportions than 2 grams of paper to 200 cc of solution were not tried. It is probable, however, that a further increase in the amount of paper used would still further diminish the fixation of free nitrogen. Now, since cellulose is rather insoluble in water, it seems strange that the presence of a mass of inert material should unfavorably influence the development of *A. vinelandii.* There is a possibility, however, that the depressing effect of the larger quantities of cellulose are due to the combined nitrogen contained in it. Reference has already been made to Winogradsky's experiment, where his *Clostridium pasteurianum* actually caused a loss of nitrogen from the solution when the amount of combined nitrogen added exceeded a certain proportion of the amount of sugar in solution. Also Beijerinck and Van Delden and Gerlach and Vogel have observed that larger quantities of combined nitrogen discourage the fixation of free nitrogen by bacteria of the azotobacter group. It will be seen that No. 5 contained 0.46 mg of combined nitrogen in the filter paper, No. 6 contained 0.76 mg, and No. 7 1.51 mg of combined nitrogen.

SERIES X.

The arrangement of the cultures in this series was as follows:

(1) 100 cc mineral solution+1 gram filter paper.
(2) 100 cc mineral solution+1 gram filter paper +0. 1 gram mannite.
(3) 100 cc mineral solution+1 gram filter paper +0. 2 gram mannite.
(4) 100 cc mineral solution+1 gram filter paper +0. 5 gram mannite.
(5) 100 cc mineral solution+1 gram filter paper +1. 0 gram mannite.
(6) 100 cc mineral solution+1 gram filter paper +1. 5 gram mannite.

The several solutions were inoculated with *A. vinelandii* and kept in the incubator for 12 days. At the end of that time the cultures were analysed and the following amounts of nitrogen were found:

No.	Ammonium hydrate (0.00302 gram N. per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.40	1.21	....
2	.75	2.26	1.05
3	.95	2.87	1.66
4	1.40	4.23	3.02
5	1.95	5.89	4.68
6	2.00	6.04	4.83

The greatest proportionate fixation took place in Nos. 2 and 4, where 1 gram and 5 grams, respectively, of mannite per liter were used. This would mean, in the latter case, a fixation of 6.04 mg of nitrogen per gram of mannite consumed. As the amount of mannite increased from 0.5 gram to 1 gram—that is, from 5 to 10 grams per liter—the yield of combined nitrogen also increased, but not correspondingly. While the increase of mannite was 100 per cent, the increase in the amount of nitrogen fixed was only 53 per cent. A further increase of the amount of mannite in solution—namely, to 15 grams per liter—was followed by a further, but not proportionate increase in the amount of nitrogen fixed. In fact, the increase here was but slight. Evidently the increasing amount of organic substance finally begins to retard the fixation of gaseous nitrogen and, as was noted in Series IX, there is a limit not only for the soluble nonnitrogenous organic material, but also for the insoluble nitrogen-poor substance, as represented here by the cellulose of the filter paper.

SYMBIOTIC FIXATION BY A. VINELANDII AND B. 30.

The fixation of nitrogen by *A. vinelandii* was stimulated by the presence in the culture solution of a small bacillus described elsewhere,[a] and designated by the laboratory number *B. 30*. Nitrogen determinations showed that this organism can fix for itself only slight quantities of free nitrogen, and it was naturally of great interest to decide just to what extent the latter contributed to the fixation of atmospheric nitrogen when in symbiotic growth with *A. vinelandii*. A number of experiments were carried out, which show very definitely that in the presence of *B. 30*, *A. vinelandii* is capable of fixing much larger quantities of nitrogen than when growing alone.

SERIES XIV.

This series was arranged to complete Series X, and is presented here accordingly. The arrangement was as follows:

(1) 100 cc mineral solution+1 gram filter paper.
(2) 100 cc mineral solution+1 gram filter paper +0. 1 gram mannite.
(3) 100 cc mineral solution+1 gram filter paper +0. 2 gram mannite.
(4) 100 cc mineral solution+1 gram filter paper +0. 5 gram mannite.
(5) 100 cc mineral solution+1 gram filter paper +1. 0 gram mannite.
(6) 100 cc mineral solution+1 gram filter paper +1. 5 gram mannite.
(7) 100 cc mineral solution+1 gram filter paper.
(8) 100 cc mineral solution+1 gram filter paper +0. 1 gram mannite.
(9) 100 cc mineral solution+1 gram filter paper +0. 2 gram mannite.
(10) 100 cc mineral solution+1 gram filter paper +0. 5 gram mannite.
(11) 100 cc mineral solution+1 gram filter paper +1. 0 gram mannite.
(12) 100 cc mineral solution+1 gram filter paper +1. 5 gram mannite.

Nos. 1 to 6, inclusive, were inoculated with *A. vinelandii;* Nos. 7 to 12, inclusive, were inoculated with *A. vinelandii* + *B. 30*. The cultures were kept in the incubator at 28° C., and were analyzed at the end of twelve days. The following amounts of nitrogen were found:

No.	Ammonium hydrate (0.00302 gram N. per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.40	1.21	....
2	.75	2.26	1.05
3	.95	2.87	1.66
4	1.40	4.23	3.02
5	1.95	5.89	4.68
6	2.00	6.04	4.83
7	.40	1.21	....
8	.90	2.72	1.51
9	1.30	3.93	2.72
10	2.40	7.25	6.04
11	3.25	9.81	8.60
12	3.20	9.67	8.46

The results obtained in solutions Nos. 1 to 6 were discussed under Series X. It remains now to compare the second half of this series with the first half of it. It will be seen that in every case where *B. 30* was inoculated together with *A. vinelandii* there was increase in the amount fixed over and above that yielded by *A. vinelandii* alone. Culture solution No. 7 should be excepted, of course, for no appreciable fixation of nitrogen took place in that case. In order to bring out these relations still more clearly, the corresponding yields are arranged parallel to one another.

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[a] J. G. Lipman, Nitrogen Fixing Bacteria, Doctor's Thesis, Cornell University, June, 1903.

No.	Fixation by *A. vinelandii.*	No.	Fixation by *A. vinelandii*+*B. 30.*	Increase.
	mg.		*mg.*	*Per cent.*
2	1.05	8	1.51	43.8
3	1.66	9	2.72	63.8
4	3.02	10	6.04	100.0
5	4.68	11	8.60	83.7
6	4.83	12	8.46	75.1

The greatest proportionate fixation was in Nos. 2 and 8, respectively. As the amount of mannite increased, the proportionate fixation decreased. There were fixed, per gram of mannite consumed, the following quantities of nitrogen:

	Mg.		*Mg.*
No. 2	10.50	No. 8	15.10
No. 3	8.30	No. 9	13.60
No. 4	6.04	No 10	12.08
No. 5	4.68	No. 11	8.60
No. 6	3.22	No. 12	5.64

It is but natural that with a smaller quantity of food in solution the bacteria should utilize it more thoroughly, and thus reduce the waste to a minimum. The experiment of Gerlach and Vogel, cited above, confirms the results presented here. There is a very close analogy in this case to the utilization of combined nitrogen in the soil by higher plants. The greater the amount of it there is present in the soil, the slighter the proportionate utilization of it within given limits. In this case the presence of *B. 30* enabled a more economical use of the organic substance in solution; nevertheless, even with the two organisms working together, there was a gradual decrease in the proportionate amount of nitrogen fixed. On the average, the presence of *B. 30* enabled *A. vinelandii* to fix 73.3 per cent more nitrogen. The minimum is 43.8 per cent, in No. 8, and the maximum 100 per cent, in No. 10.

Repeated experiments which it would be superfluous to give here in detail still further emphasized the facts noted above. They all show that aside from the increased fixation, the presence of *B. 30* also enables *A. vinelandii* to withstand better the presence of excessive quantities of organic matter. Hence, it would appear that the accumulation of large quantities of organic matter in the soil, be it rich or poor in nitrogen, unfavorably affects the development of azotobacter bacteria. When there is an accumulation of large quantities of nitrogen-rich substance, the growth of these organisms practically stops, when there is an accumulation of nitrogen-poor organic substance, the growth of these organisms is retarded. But since in all arable soils many bacterial species act and interact on one another, and the development of any particular combination may become prominent at one time and of another at some other time, it is clear that the conditions of fixation in the soil are perforce somewhat different from those existing in artificial cultures. None the less, they show unequivocally that, for its best work, *A. vinelandii*, and undoubtedly also other members of the azotobacter group, must depend on the cooperation of other organisms.

SERIES XVI.

This series is complimentary to Series XVII, and was carried out with a pure culture of *A. vinelandii.* It was intended to study the effect on fixation of a large exposed surface. For this reason the mannite solution employed was placed in large crystallizing dishes covered in the manner of ordinary Petri dishes. The arrangement in this series was as follows:

(1) Large Erlenmyer flask, 300 cc mannite solution, sterile.
(2) Large Petri dish, 300 cc mannite solution, *A. vinelandii.*
(3) Large Petri dish, 500 cc mannite solution, *A. vinelandii.*

The internal diameter of the dish in No. 2 was 27 cm. and in No. 3, 29 cm. The mannite solution contained 15 grams of mannite per liter; hence Nos. 1 and 2 contained 4.5 grams of mannite each, and No. 3 contained 7.5 grams of mannite. After sterilization, cooling, and inoculation in the usual manner, the dishes were placed in the incubator at a temperature of 28° C. In both of the inoculated dishes growth was apparent in twenty-four hours. In forty-eight hours there was the development of the bright yellow pigment observed in all mannite cultures of *A. vinelandii*, and of the characteristic membrane on the surface. As it grew older the membrane became gradually thicker. On examination under the microscope it was found that *A. vinelandii* alone was present, and hence there had been no contamination from the outside. The contents of the dishes and of the flask were analyzed at the end of six days, and the following amounts of nitrogen were found:

No.	Ammonium hydrate (0.00302 gram N. per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.20	0.60	....
2	9.55	28.84	28.24
3	17.40	52.55	51.95

During digestion part of the solution in No. 2 was lost, and the amount given here is undoubtedly somewhat too low. While the loss is not believed to have been very considerable, it amounted to 2 or 3 mg. Leaving No. 2 out of consideration, we note that there were 51.95 mg of nitrogen fixed in No. 3; hence there was a fixation of 6.8 mg of nitrogen for every gram of mannite consumed. This is a very considerable quantity for a period of six days, and by a pure culture of *A. vinelandii*. These results were obtained with a culture which had been reinoculated into artificial solutions a considerable number of times, and they show that *A. vinelandii* does not lose its nitrogen-fixing power, as was reported in the case of other nitrogen-fixing organisms.

SERIES XVII.

The culture solutions in this series were arranged as follows:

(1) 200 cc mannite solution in Erlenmeyer flask + 1 gram filter paper.
(2) 200 cc mannite solution in large Petri dish + 1 gram filter paper.
(3) 200 cc mannite solution in large Petri dish + 1 gram filter paper.

No. 1 was left sterile, No. 2 was inoculated with *A. vinelandii*, and No. 3 with *A. vinelandii* + *B. 30.* There was also added in each case 1 gram of calcium carbonate. At the end of 10 days the several solutions were analyzed and the following amounts of nitrogen were found:

No.	Ammonium hydrate (0.00302 gram N. per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.15	0.45	....
2	9.90	29.90	29.45
3	10.15	30.65	30.20

The solutions contained at the beginning of the experiment 3.0 grams of mannite in each case, so that there were fixed in No. 2, 9.81 mg of nitrogen for every gram of mannite consumed. Similarly, there were fixed in No. 3, 10.07 mg of nitrogen for every gram of mannite consumed. These are the highest yields thus far obtained in the 15 grams of mannite per liter. The presence of *B. 30* increased the yield also in this case, although the increase was rather small.

FIXATION OF NITROGEN BY A. VINELANDII IN THE PRESENCE OF OTHER SOIL BACTERIA.

These studies include experiments with a number of organisms. Their purpose was to determine whether there are bacteria other than *B. 30* that would favor the fixation of free nitrogen by *A. vinelandii.*

SERIES XVIII.

The solutions employed in this series contained, besides the customary amounts of the mineral salts, either 20 grams of mannite, or of glucose, per liter of solution. Two hundred cc portions of these solutions were distributed in Kjeldahl flasks, with the addition in each case of 1 gram of calcium carbonate. The organisms used in this series were *A. vinelandii*, *B. pyocyaneus*, *B. 31*, *B. 30*, *B. 31* var., and *B. 32*. The arrangement of the experiment was as follows:

(1) Glucose solution.	Sterile.
(2) Mannite solution.	
(3) Glucose solution.	*A. vinelandii.*
(4) Mannite solution.	
(5) Glucose solution.	*A. vinelandii* + *B. pyocyaneus.*
(6) Mannite solution.	
(7) Glucose solution.	*A. vinelandii* + *B. 31.*
(8) Mannite solution.	
(9) Glucose solution.	*A. vinelandii* + *B. 30.*
(10) Mannite solution.	
(11) Glucose solution.	*A. vinelandii* + *B. 31* var.
(12) Mannite solution.	
(13) Glucose solution.	*A. vinelandii* + *B. 32.*
(14) Mannite solution.	

At the end of 8 days the cultures were analyzed and the following amounts of nitrogen found:

No.	Ammonium hydrate (0.003038 gram N. per cubic centimeter).	Nitrogen found.	Nitrogen fixed.
	cc.	*mg.*	*mg.*
1	0.25	0.76	....
2	0.15	0.45	....
3	1.35	4.10	3.34
4	1.45	4.40	3.95
5	1.55	4.71	3.95
6	0.90	2.73	2.28
7	1.55	4.71	3.95
8	1.55	4.71	4.26
9	3.25	9.87	9.11
10	3.55	10.78	10.33
11	1.55	4.71	3.95
12	1.85	5.62	5.17
13	1.35	4.10	3.34
14	1.95	5.92	5.47

These results show that, with one exception, there was more nitrogen fixed in the mannite solutions than there was in the glucose solutions. They also show that

B. 30 was the only organism of those tried in the series which increased the fixation of nitrogen by *A. vinelandii* to any considerable extent. Thus *A. vinelandii* alone fixed in glucose 3.34 mg of nitrogen, and in mannite 3.95 mg of nitrogen. In the presence of *B. 30* the corresponding amounts were 9.11 mg and 10.33 mg—more than 2.5 times as much. It also seems that in Nos. 12 and 14 there was a marked increase in the amount of nitrogen fixed, due to the presence in No. 12 of *B. 31* var., and in No. 14 of *B. 32*. Further combinations of *A. vinelandii* with one or more of the other organisms showed that none of them gave any considerable increase beyond that given by *B. 30*.

The experiments presented here are sufficient to show to what extent the members of the azotobacter group are capable of fixing atmospheric nitrogen in artificial solutions. There are many other experiments at hand representing hundreds of analyses which are confirmatory of the above, and it is now proposed to extend the experimental work to actual soil conditions, in order to discover whether the bacteria of this group may be utilized to replace the nitrogen removed from the soil by crops.

Mr. WITHERS. I have a resolution which I shall be glad to have referred to the committee on recommendations of the referee on soils. It is as follows:

Resolved, That the referee on soils be requested to test if possible the value of proposed methods for comparing the ability of different soils to support the growth of nitrifying organisms when the conditions are identical as to temperature, moisture, number of germs, etc.

The PRESIDENT. If there is no objection this resolution will be referred to the committee on recommendations on soils.

Mr. WHEELER. I have no formal paper, Mr. President, but I wish to call attention to a new indictment that we have to bring against ignited iron and aluminum phosphate or roasted "redondite." This redondite contains from 35 to 40 per cent of reverted phosphoric acid and hence is a convenient material with which to raise the percentage of "available" phosphoric acid in mixed fertilizers. Persons engaged in the fertilizer business say that it is used extensively, and yet this phosphate when employed on acid soil has very little value, and as concerns its after effect is practically worthless. This is a very serious indictment to bring against a material which contains from 35 to 40 per cent of available or reverted phosphoric acid. Phosphate, however, when applied upon a limed soil is much more valuable. The limed soil used was probably not limed heavily enough to bring all of the organic acids into union with lime, though it may have been temporarily neutral or alkaline to litmus paper. It was sufficiently limed, however, to produce all ordinary crops satisfactorily.

Upon this limed soil and with certain crops this phosphate was nearly equal, in the results produced, to some of the most valuable phosphates. This was true of grass for a period of about four years. But when used with many of the hoed crops, particularly with turnips, beets, squashes, lettuce, etc., this material was of small value, even upon the limed soil, as compared with basic slag-meal and finely ground bone, either acidulated or unacidulated, and with other phosphates. In Rhode Island we are face to face with this proposition—either our soils must be put in a condition such that this material will be effective or our method of analysis must be modified so that we may discriminate against this particular phosphate. As the chemists can not compel farmers to lime their acid soils it seems that we should modify our methods or possibly determine the alumina in the goods as an indication of the presence of this phosphate. The leading facts concerning this matter as ascertained to date may be summarized as follows:

> *Panicum crus-galli*, common millet, and a few other plants, seem to have exceptional ability to use ignited aluminum phosphate on both limed and unlimed soil. Its efficiency for these crops is increased, nevertheless, by liming. For grass on limed soil, its efficiency was very close to that of some of the best phosphates, but upon acid soil its relative and absolute efficiency were much less. For many of the hoed crops, Swedish turnips, beets, flat turnips, squashes, lettuce, and others, its after effect, at least upon acid soil, amounts to practically nothing. Its after effect upon limed soil is relatively better than upon the unlimed soil, yet the crops, even then, were poor compared with those produced by the lime phosphates, acidulated or unacidulated. Those plants which seem to make the best use of ignited aluminum phosphate on unlimed soil are generally those least affected by acidity. This may be merely because they are able to exist under such acid conditions rather than on account of any special ability to feed upon this particular phosphate.

This is a brief summary of the conclusions reached and in view of these facts I wish to bring before the association as forcibly as possible the desirability of taking under immediate consideration our methods for determining reverted phosphoric acid in commercial fertilizers. This particular material is introduced very largely into our commercial fertilizers not only by those who control the process of making it, but also by others, and one of the principal reasons for its employment is that it produces a fertilizer which drills readily, a feature that pleases the farmer. I wish that some member of this association who lives on an alkaline soil would test this same material for a series of years and find out if under such soil conditions this phosphate. is as effective as the other phosphates even for such crops as turnips and beets.

Mr. McDONNELL. It is interesting to note in connection with Mr. Wheeler's remarks that the fact that the aluminum phosphates are more available in alkaline soils agrees with the fact that these phosphates are more soluble in the citrate solution when it is slightly alkaline than when it is slightly acid.

Mr. WHEELER. Referring to some remarks previously made I wish to give a caution against using enough lime to bring all the organic acids into combination at the outset, because I believe that practically all the benefit to be derived from liming may be secured at once by using less than that amount even on very acid soils. At least for a few years the use of much less lime than is necessary to permanently neutralize the total acidity of the soil will insure good crops and the agricultural chemist must consider the economical aspect of such matters. While it might be well to neutralize all the acidity at once, or, in other words, combine if possible all of the organic acids and other acid substances with lime, it would be quite expensive, and hence it is better to effect this only partially at first and later to neutralize completely.

At 4.20 p. m. the association adjourned.

THIRD DAY.

SATURDAY—MORNING SESSION.

The convention was called to order by the president at 9.30 a. m. The report on phosphoric acid was called for and in the absence of the referee, who had sent no formal report but in a letter to the secretary had made certain observations relative to the work, the following informal report was submitted by the secretary:

REPORT ON PHOSPHORIC ACID.

By B. H. Hite, *Referee.*

(1) The association made no recommendations or suggestions in regard to the determination of phosphoric acid, which the referee thinks was wise. The present method for determining phosphoric acid meets every requirement, while the shortcomings in the methods for the determination of other constituents could well consume all the time at the disposal of the association for cooperative work.

(2) The associate referee was anxious to test yet other modifications of the volumetric method as applied to the determination of phosphoric acid in soils. The referee did not care to take part in any further cooperative work on this subject, and so instructed the associate to take full charge of that investigation, sending out his own samples, etc.

(3) The referee was anxious to do something toward improving existing methods and turned his attention to certain modifications of well-known methods involving the weighing of the yellow precipitate. In the hands of the referee the methods gave most excellent results, but when tried by other analysts indifferent results were obtained. The work was again taken up by the referee, and it is believed that the cause of the varying results can be located, but it is not deemed advisable to present the work to the association until more time has been given to it.

Mr. Van Slyke. In this connection I wish to submit by title a paper on "The Determination of Organic and Inorganic Phosphorus in Vegetable and Animal Materials," by E. B. Hart and W. H. Andrews, of the Geneva station.[a]

Mr. Williams. I have a report on phosphoric acid to submit to the association.

COMPARISON OF THE VOLUMETRIC AND GRAVIMETRIC METHODS IN DETERMINING TOTAL PHOSPHORIC ACID IN SOILS.

By C. B. Williams, *Associate referee.*

The work of the associate referee on phosphoric acid was confined this year entirely in testing the relative efficiency of the volumetric and gravimetric methods in the determination of total phosphoric acid in four soils, two from North Carolina and one each from California and Illinois. This was undertaken because it is believed that a knowledge of the total quantity of plant food in any soil will afford a more complete understanding of its potentialities, cultural, and fertilizer requirements than results obtained by any other method. A circular letter was sent to the chief chemists of all agricultural experiment stations asking cooperation in the work for this year. Samples were sent to the seven who replied favorably, but analytical data have been supplied only by three analysts.

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[a] This paper may be found in Bulletin No. 238 of the New York (Geneva) station, and in the American Chemical Journal, vol. 30, No. 6, December, 1903.

DIRECTIONS FOR WORK ON PHOSPHORIC ACID SAMPLES FOR 1903.

I.—MOISTURE.

Use the official method described in Bulletin 46, revised edition, Bureau of Chemistry, U. S. Department of Agriculture, page 71.

II.—METHOD OF SOLUTION.

Place 3 grams of soil in a platinum dish and ignite until organic matter has been destroyed, then treat three times with from 3 to 4 cc of hydrofluoric acid, evaporating to dryness each time on a water bath, using a platinum rod to stir upon each addition of acid. Mix the residue thus obtained with 10 grams of a mixture of equal parts of sodium and potassium carbonates (free from phosphoric acid), and reduce in an agate mortar to a fine powder, after which heat over a blast lamp, gently at first, until the mass has completely agglutinated, when stronger heat should be turned on and continued until calm fusion is attained. Then cool, place the dish and its contents in a beaker and add sufficient (1:1) hydrochloric acid to cover the dish. Place on a water bath and digest until the mass has thoroughly loosened from the dish, after which it is removed. Evaporate to dryness on a water bath and thoroughly dehydrate the silica present by finishing the heating in an air bath at 110° C. for 4 hours. Take up with dilute hydrochloric acid and digest on water bath for 30 minutes, after which filter from silica, and wash thoroughly to remove traces of phosphoric acid. To the filtrate add sufficient nitric acid to liberate all chlorids and evaporate the solution to a volume of about 40 cc. Then neutralize the excess of nitric acid with ammonia and add 12 grams of ammonium nitrate.

After effecting solution as indicated above, determine phosphoric acid in the four soil samples by the following methods:

III.—GRAVIMETRIC METHOD.

The regular official method (Bul. 46, revised edition, Bureau of Chemistry, U. S. Department of Agriculture, p. 74).

IV.—VOLUMETRIC METHOD.

Cool, add 30 cc of recently filtered molybdic solution, and securely stopper (antimony rubber stoppers have been found most satisfactory). Shake the flask in a shaking machine or by hand for 30 minutes, after which remove and filter through carbon filters by means of suction, using asbestos mats.

After thoroughly transferring the ammonium phospho-molybdate precipitates and washing out the flasks onto the asbestos filters, give the precipitates six more washings each. Then remove the stoppers from the pressure flasks with the small ends of the carbon filters still stuck through them and hold upright over the sink, washing the outsides free from acid with distilled water. Reverse the carbon filters into the mouths of the flasks which originally contained the precipitates, still holding the small stems, and by means of the copper wires that extend beyond the small ends of the carbon filters push out the disks, asbestos, and precipitates into the flasks and wash the disks and inside of carbon filters carefully with a small stream of water. Dissolve the precipitates in a slight excess of standard alkali, add a few drops of phenolphthalein solution to each and titrate back with standard (nitric) acid, using a long stirring rod to thoroughly agitate during the operation.[a]

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 46, pp. 13–14; J. Amer. Chem. Soc. 28: 8.

(*a*) *Apparatus.*

1. *Precipitating flask.*—This consists of a 500 cc Erlenmeyer flask with neck 40 mm inside diameter and rubber stopper to fit.

2. *Filtering device.*—Through a rubber stopper in a 16 ounce pressure bottle of Erlenmeyer form is passed the small end of a carbon filter; in the bottom of this is placed a perforated porcelain plate or disk, to which is rigidly fastened a No. 19 copper wire about 25 cm long that projects downward into the pressure bottle. The disk is covered with a thin layer of asbestos prepared according to Gooch.

3. *Shaking machine.*—Use the apparatus devised by Wagner, except that the caps that fit over the tops of the flasks are made larger. This machine holds ten flasks and can be revolved either by hand or motor. A speed of 40 to 50 revolutions per minute should be maintained, as this is the velocity that has been found to give the maximum agitating efficiency.

(*b*) *Preparation of reagents.*

The reagents are as indicated in the official methods, except that the strength of the acid and alkali is one-half the strength there prescribed; i. e., one cc will be equal to one-half milligram phosphoric acid (0.01 per cent phosphoric acid on a basis of 5 grams of soil).

1. *Distilled water.*—All washing and diluting should be with distilled water. Distilled water obtained from water containing a large amount of bicarbonates is liable to contain considerable carbon dioxid in solution. This water can be used in washing the ammonium phosphomolybdate, but should be used neither in transferring the precipitate nor in diluting before titration. Boiling the water prior to using will obviate this difficulty.

V. REPORTING RESULTS.

All results should be tabulated, after calculating to water-free basis, and sent with complete notes to the associate referee at least thirty days prior to the meeting.

Charles B. Williams,
Associate Referee.

Description of Soils.

No. 1.—This soil is a mellow, fine, sandy loam of sedimentary origin, underlaid by a moderately retentive clay subsoil at a depth of 7 inches. It was taken from the middle of the fertilizer test plats of corn in center of third series of unfertilized plats at the Edgecombe test farm of the North Carolina department of agriculture. This soil represents a very extensive type in the eastern part of the State that is especially adapted to the production of cotton, corn, peanuts, truck, and bright-leaf tobacco. This land has been in cultivation for about 75 years and has been principally devoted to cotton, corn, oats, wheat, tobacco, and clover. Its original forest growth was pine, oak, and sweet and black gum.

No. 2.—Heavy red clay loam, designated by the Bureau of Soils as Cecil clay, was taken at a depth of 8 inches from plat No. 5, second series of unfertilized corn plats of the Statesville test farm of the North Carolina department of agriculture. This soil is of residual origin, "formed in situ, by the long continued processes of decomposition, from a number of rock types distinct in their physical and mineralogical characters. The soil has been derived from nearly all of the rocks occurring in this section, mainly hornblende gneiss, micaceous schists, coarse-grained granites, and, to a less extent, soapstone or steatite."

Although the soil is underlaid by a stiff tenacious red clay subsoil to a depth of several feet, it possesses good natural drainage. It is a strong, valuable soil and is especially adapted to the growing of grains, grasses, and clover. Cotton will grow upon it, but it is not so well suited to this crop as to the ones first mentioned. The original native growth was principally black-jack oak, pine, and black and sweet gum.

No. 3.—This is a dark soil, containing considerable humus to the depth of 7 inches, taken from the terrace or second bottom land along the Du Page River in Du Page County, Ill. It lies from 20 to 30 feet above high-water mark and probably has not been overflowed for centuries, although it may have at one time been bottom land of the Du Page River. It is made up largely of glacial drift, somewhat modified by the underlying limestone deposits. It belongs to one of the richest classes of agricultural soils of the State of Illinois. The land has been in alfalfa for three years and has produced as high as 10½ tons of alfalfa hay per acre in a single season. Under favorable seasonal conditions, says Mr. Cyril G. Hopkins, who furnished the soil, it will undoubtedly produce from 90 to 100 bushels of corn per acre.

No. 4.—This sample, furnished by Director E. W. Hilgard, is a fine, heavy, dark clay soil, collected from near the town of Arroyo Grande, in the valley of Arroyo Grande, in the southern portion of San Luis Obispo County, Cal. This soil is very rich in plant food and is especially noted among seedsmen as one "capable of producing vegetable seeds of maximum value." Over one-half of this soil is silt, and possesses a saturation point by volume of 67 per cent and by weight of 77 per cent. The mechanical and chemical analyses of this soil (No. 2061) will be found on page 178 of the Report of the California Agricultural Experiment Station for the years ending 1898–1901, Part II.

RESULTS OF WORK.

The analytical results, given in the table, are calculated to water-free substance, and hence are on a strictly comparable basis.

Determination of total phosphoric acid by two methods in four (calculated to water-free substance) soils.

Analyst.	No. 1.		No. 2.		No. 3.		No. 4.	
	Volumetric.	Gravimetric.	Volumetric.	Gravimetric.	Volumetric.	Gravimetric.	Volumetric.	Gravimetric.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
F. P. Veitch, Washington, D. C.	0.043	0.037	0.086	0.077	0.272	0.322	0.468	0.529
W. G. Haywood, Raleigh, N. C.	....	....	....	....	.289	....	.520	....
	....	....	....	....	.308	.311	.499	.517
C. B. Williams, Raleigh, N. C.	....	.093	....	.079	.305	.336	.501	.496
	.044	.096	.076	.080	.302	.296	.492	.503
J. H. Pettit, Urbana, Ill.[a]	.050	.048	.049	.049	.276	.274	.495	.500
Average	.043	.066	.081	.079	.291	.316	.492	.515

[a] Received too late to be included in averages; see comments by analyst.

COMMENTS BY ANALYSTS.

F. P. Veitch.—The instructions were followed in all essential particulars. In the execution of the volumetric method the yellow precipitate was filtered and washed free of acid on the ordinary filter paper and funnel. No shaker was used, but the precipitate was allowed to stand four hours at 50° C. before filtering. In regular work with the gravimetric method it is my practice, after dissolving in ammonia, to reprecipitate with nitric acid and more molybdate and complete the determination as usual, washing the precipitate very thoroughly with water. In routine work we always use the volumetric method on soils.

Harry Snyder.—One set of determinations was made on the soil samples, but for lack of time have not been able to duplicate the results. Mr. Hummel, the assistant chemist, who did the work, reports that the results by the gravimetric method are slightly higher than those obtained by the volumetric. In some soil work we have divided the solution, used part for the gravimetric and part for the volumetric determination, and then dissolved the gravimetric precipitate after weighing, and determined the phosphoric acid volumetrically. Determinations made in this way compare quite closely. I think the difficulty is due to the presence of a little alumina and other compounds in the final precipitate, and on that account the results by the volumetric method are more satisfactory than those by the ordinary gravimetric process.

W. G. Haywood.—The results on soils Nos. 1 and 2 were not altogether satisfactory, and no report is made on them, as sufficient time was not available to make further determinations. Considerable difficulty was experienced with Nos. 1 and 2 in keeping the phosphoric acid from going out of solution while being evaporated with nitric acid prior to adding molybdic solution; with soils Nos. 3 and 4 no trouble was encountered. Of the two methods, I prefer the volumetric, as it is easier to manipulate than the gravimetric and gives equally as good results.

J. H. Pettit.—I had difficulty in running the method suggested for getting the phosphoric acid into solution. In our regular work on soils we use the 10-hour digestion method for obtaining the soil solution. For the determination of phosphoric acid in this solution an aliquot portion is concentrated to 25 cc, neutralized with ammonia and cleared up with nitric acid. Then heat to 40°, precipitate with molybdic solution and let stand over night. Filter, wash, dissolve in standard potassium hydroxid, and titrate the excess of the latter with standard nitric acid. This is practically the same method as that outlined by the association and has been checked up with the gravimetric method. Further, for my personal satisfaction, I used the 10-hour digestion on the soils sent out by the referee, intending to compare the results obtained with those given by the fusion method. In these solutions the phosphoric acid was determined both by our method and by the suggested shaking method. The results are as follows:

Comparison of phosphoric acid determinations in soils by different methods, under varying conditions.

Soil No.	Gravimetric method precipitated at—			Volumetric method, shook 30 minutes.
	40° C.	65° C.	80° C.	
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*
883	0.057	0.055	0.055	0.052
884	.065	.065	.063	.055
885	.103	.104	.103	.098
886	.081	.082	.080	.075
887	.095	.095	.096	.088

DISCUSSION OF RESULTS AND METHODS.

By a study of the results in the table it will be found that, with the exception of soil No. 2, the average results on total phosphoric acid are higher by the gravimetric than by the volumetric method. These divergences are due most probably to the small amounts of iron, alumina, or silica, or all three, carried down with the white precipitates when magnesia mixture is added, as pointed out by the associate referee in the Journal of the American Chemical Society[a] and by others elsewhere. For this reason, and for the further reason that the volumetric method precipitated all phosphoric acid in the four official samples, and in about three hundred other determinations of soil samples made by the associate referee, it is thought that the volumetric method is preferable to the gravimetric in the determination of total phosphoric acid in soils. Also the volumetric is much shorter than the gravimetric method.

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[a] Vol. 25, p. 493.

RECOMMENDATIONS.

It is recommended:

(1) That the volumetric method for the determination of total phosphoric acid in soils be made an optional method.

(2) That a study of different solvents for total phosphoric acid in soils be taken up for investigation next year.

The PRESIDENT. Are there any remarks to be made on phosphoric acid?

Mr. ROBISON. We have had trouble in Michigan with a certain class of phosphates which when treated with a sufficient amount of citrate of ammonia go completely into solution. Some experiments show that from a half of 1 per cent to 1 per cent is insoluble by the official method when 2 grams are used, whereas if 1 gram is used there will remain practically no phosphate undissolved. With a precipitated phosphate that is put out by the Michigan Carbon Works of Detroit, if we use 2 grams and 100 cc of citrate of ammonia of 1.09 specific gravity varying percentages are undissolved. The manufacturers regard this as quite an important point, as they think the present method misrepresents their goods.

The PRESIDENT. This brings up an old question with which we have been having trouble for some years and I do not know of any disposition that can be made of the matter unless this single substance be singled out and treated differently from any other phosphate. The question might be referred to the referee for investigation as to what action shall be taken.

Mr. HUSTON. For the consolation of our friend from Michigan I would say that I have a beautiful curve on this subject which I have had for about ten years. The material has not been published except in a fragmentary way, but I have quite complete data in the laboratory and shall be glad to give any information that I can on the subject. So far as making this a separate subject is concerned I would say that it really occupies a peculiar position as precipitated phosphate is probably as near reverted phosphoric acid as anything found in the trade. The method of preparation is exactly that of theoretically reverted phosphoric acid, I understand, so it practically is reverted.

Mr. WILEY. I wish to submit the following letter received from Mr. Hilgard of the California station under date of October 28, 1903:

> At a late meeting of the Association of Official Agricultural Chemists the subject of an official method for the determination of available phosphoric acid in Thomas slag was dropped, on the ground that not a sufficient amount of the slag was being used in the United States; but now the use of the slag in California is extending rapidly, amounting last year to over 1,500 tons, and as our fertilizer law requires us to use the official method, we are in a predicament. As I understand it, the citrate method has

> by default remained the official one, while in Europe it is almost universally discarded for the citric acid method, which corresponds more nearly to actual experience. Now, the slag comes with a certificate of available phosphoric acid according to the European method, while we are supposed to use here the one that has been discarded, and thus put the importer in jeopardy of the law. I would suggest, therefore, that this matter be taken up again at the coming meeting and referred to the proper committee for consideration.
>
> The reason for recommending the use of Thomas slag on our soils is simply this: The arid soils are so universally rich in lime carbonate that reversion of superphosphate occurs within the first 3 or 4 inches of the surface soil, hence to bring the phosphate within reach of the roots of our deep-feeding plants it must be deeply plowed in to be effective. But the same plowing will also make the slag phosphate effective, and that being the cheaper source I recommend its use on the basis of European experience. * * * Moreover, it is especially adapted to the limonite soils of the Hawaiian Islands, in which the soluble superphosphate rapidly reverts to the same inert condition in which it is already contained, and very abundantly, in the basaltic soils of Oahu and Hawaii. Quite similar soils also occur in California and Oregon, so the question of the Thomas slag is of considerable interest to the Pacific coast and should be taken up and settled by the association.

I wrote to Mr. Hilgard, citing the proceedings of this association where the matter had been discussed, and quoted the suggestion made by a gentleman who has had a great deal of experience in this matter, to the effect that the establishment of fineness for the basic slags together with an estimate of the total phosphoric acid might be satisfactory to all concerned. Professor Hilgard telegraphed as follows:

> Our station fully agrees to the standard of fineness and total phosphoric acid for Thomas slag.

I think it might be well for the referee to be asked to again consider the advisability of establishing some special method of valuing these slags. I know the danger of permitting a valuation of this kind, but it is just as fair to the slag as it is to the bone, because we do determine the total phosphoric acid in bone. Whether the State allows it in trade values or not is another question, but the official method does determine phosphoric acid in bone. We also determine the total phosphoric acid in all phosphoric fertilizers, and there is no reason why that plan should not be adopted for basic slag. Then if the degree of fineness be considered in conjunction with the total phosphoric acid as a factor in estimating its availability it appears that a conclusion satisfactory to all concerned will be reached.

Since this meeting began Mr. Huston has again called my attention to the fact, which he observed, and was one of the first to observe in his earlier service under my direction at Purdue, namely, that fine-ground bone goes into progressive solution in the ammonia citrate solution with which reverted phosphoric acid is estimated, and the amount which goes into solution is a factor or function determined by fineness, the strength of the solution, the temperature, and the time of digestion. So that any amount, almost, of the finely ground bone could be rendered available by this treatment. Now, the same thing is true of basic slag. By adopting the European citric-acid method as the standard for valuation you can practically, by continuing the application or varying the strength or temperature, bring the whole of the basic slag into solution and render it thus apparently available. Or, by varying the conditions, you may reverse the reaction to a certain extent.

It is also well known that basic slag is far more available for plant food, as a rule, than an equal amount of phosphoric acid in the form of tricalcium phosphate undissolved, although both may be equally insoluble in water, the basic slag having a different chemical composition and therefore being more available for the nourishment of a plant. In connection with one of my assistants, I presented to this association several years ago some papers on the determination of the quantity of phosphoric slag really present in the form of tetra-calcium phosphate and the system of its crystallization. When we consider the composition of a basic slag and remember that it is a mixture of free lime, iron to some extent, tetra-calcium phosphate, with perhaps a trace of tricalcium phosphate, and many other substances, it is seen that we have a very complicated material with which to deal. Nevertheless, though the consideration of it was formerly dropped by the association because it was not coming into the country to any extent and was not largely used as a fertilizer, in the light of the statement made by Mr. Hilgard we must now reverse that opinion and recognize that basic slag is again becoming commercially important in this country.

I therefore move that the referee on phosphoric acid be instructed to reconsider the question of the valuation of basic phosphatic slags, especially the recommendation to establish a standard based on the total phosphoric acid and degree of fineness.

The PRESIDENT. Perhaps Mr. Wiley would include the precipitated and the Redonda phosphates.

Mr. WILEY. I have no objection to that.

Mr. HUSTON. There is one point in connection with these slags, which seems to have had the special attention of our friends in Europe who determine availability by citric acid alone, that we ought to consider. While the tricalcium phosphate goes into solution progressively under different conditions of temperature with basic slag, the amount of phosphoric acid going into solution increases for a time with citric acid. It then decreases, and if the action of 1 or 2 per cent citric acid is continued long enough a point is reached where there will be a very large amount of lime, iron, manganese, and the other constituents of the slag in solution, and no phosphoric acid at all is left, it being entirely precipitated.

I do not believe that any method which under varying conditions of temperature will give a maximum at different times and finally give nothing is a suitable method for any commercial material, and it seems to me that the matter should be very seriously considered. The fact that phosphoric acid will not remain in solution in citric acid if the action be sufficiently prolonged has not received much attention. Moreover, in dealing with citrate of ammonia it must be remembered that the solution must be much more dilute, or, rather, with two grams of slag and 100 cc of liquid the amount dissolved is increased very rapidly by simply adding water. We do not make any progress of real value unless these two simple facts are considered.

Furthermore, there are other slags which you will be called upon to value later. They are not the Bessemer crucible slags, but the open-hearth slags, which are now made in very large quantities near St. Louis, and as the Bessemer ores are exhausted the phosphoric acid in these slags will rise until they finally become an article of commerce. They differ somewhat from the others in that they contain a large amount of silica, which may make trouble. Enormous quantities of these open-hearth slags will be produced before long, and some of them already contain 10 per cent of phosphoric acid, which almost places them in the commercial class.

The PRESIDENT. The resolution offered by Mr. Wiley is before the convention.

The resolution was adopted.

Mr. PENNY. I wish to offer the following resolution on soil, if it is in order:

Resolved, That the referee on soils be requested to make a thorough examination of the methods described in Bulletin 22 of the Bureau of Soils for the determination of available plant food.

Adopted.

Mr. CAMERON. Mr. President, I wish to say that a misunderstanding has evidently gone forth, and is in the minds of the majority of the gentlemen here, in regard to the purpose of Bulletin No. 22. It was not maintained in that bulletin that the procedure there described would determine the available plant food in the soil in the sense in which "available plant food" is supposed to be determined by the conventional procedures. The analytical methods used were given as an explanation of our work and an earnest of good faith. They were developed for our own particular purposes, although of course we can not have the slightest objection to anybody using them.

Our object in the beginning of the work was to develop methods which would enable us to determine exceedingly small amounts in solution and then with these methods to develop a procedure which would enable us to follow what changes might be taking place in the soil from day to day. It was found impracticable in the field work described in Bulletin No. 22 to make the examinations daily, but we did as well as we could by making them every week or ten days during the growing season.

I wish to state distinctly to this convention, so that there may be no misunderstanding, that while there is no objection to anyone trying these methods, the authors of Bulletin No. 22 do not and never have proposed a procedure for the determination of the fertility of soils. Bulletin No. 22 shows that the procedure we followed would not work; it does not determine the fertility of the soil, and in this regard is absolutely without analogy to other proposed methods, such as the methods to which Mr. Wiley has referred, those Mr. Hilgard has suggested, or the "official" method. Our methods were intended for an entirely different purpose, and the Bureau of Soils has never supposed they would be considered as proposed for the same purpose as the official method.

Now we may some time propose a method for determining fertility—Professor Whitney and I have been investigating one—but it is something entirely different in character from the work described in Bulletin No. 22. I will say once more that the procedures described in Bulletin No. 22 are not, and there is nothing in the bulletin which can be tortured into saying they are, methods for the determination of the fertility of the soil in the sense in which conventional methods are so used.

The PRESIDENT. We will now take up the report on sugar, and in justice to the referee, Mr. Tolman, I wish to say that he took up the work late in the year, at my request, upon the resignation of Mr. Spencer.

REPORT ON SUGAR.

By L. M. TOLMAN, *Referee.*

The work on sugar this year has been very limited. Mr. Munson has continued the work on reducing sugar commenced last year, and will make a report at this meeting. Mr. Davoll, associate referee on beet-sugar methods, through some misunderstanding, did not send in any report to the meeting, but has since made a few suggestions in a letter to the referee as to work for next year, which are as follows:

> I have been considering the matter of taking up several lines of investigation after the campaign is over, continuing the work on raffinose and starting others. We lay great stress upon raffinose determinations in our factory, as the carbohydrate is present to a considerable extent in Michigan beets. As the campaign progresses I shall be able to suggest some cooperation work after watching our own laboratory work during that time.
>
> Regarding additional work, I would suggest investigations upon the determinations of sucrose and raffinose in the presence of reducing sugars. J. Wortman[a] has worked out a formula, but I believe that it is not generally accepted as correct.
>
> Another thing that we need is a practical method for the direct analysis of the beet by the hot-water-digestion method in the tare room, capable of handling up to 500 samples per day, with the minimum of labor. I am inclined to favor the Sachs-Le Docte method, but look for a simplified manipulation as regards the digestion. An instantaneous cold method would do if a uniform and continuous supply of fine pulp could be had.

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[a] Spencer's Handbook, p. 110; also Zeit. Rübenzucker-Ind., 39: 766.

Mr. Munson's paper being in the nature of a report of progress, he makes no recommendations except that a continuation of the work is very desirable. It is hoped that by next year some definite results may be offered the association, which are especially needed, as at this time we have two distinct methods of procedure for the Allihn method which give different results, and yet the same tables are used.

RECOMMENDATIONS.

(1) The question of the analysis of such products as molasses has been under discussion lately, as the methods now in use require certain modifications before they can be applied, and the question has been raised as to whether it would not be well to appoint another associate referee on sugar to take up the subject of the application and modification of our present methods to apply to such products. I think that the only way in which we can accomplish much in sugar work, as in most of the other lines of work in this association, is through individual effort, and would recommend that such an associate be appointed. I believe Mr. Sawyer, who is much interested in this subject at the present time, will give an outline of the work he has been doing on molasses methods and make some suggestions as to work for next year.

(2) The international association for the unification of sugar methods has at the present time under discussion some changes in the copper solution used to determine reducing sugars, and I would recommend that the referee be instructed to communicate with the committee which has this work in charge with the idea of cooperating with them on this subject.

(3) The recommendations involving changes in official and provisional methods on sugar and several minor changes in Bulletin No. 46 which were made at the last meeting and printed in the proceedings of that year (1902) were overlooked by the committee on recommendations of referees, and should be acted upon at this meeting. (See report of Committee B, p. 230.)

The PRESIDENT. Before proceeding further with the discussion of the sugar report, I wish to introduce Doctor Needham, of the Columbian University.

ADDRESS BY PRESIDENT NEEDHAM, COLUMBIAN UNIVERSITY.

Mr. PRESIDENT AND GENTLEMEN: It gives me great pleasure on behalf of the University to extend a cordial greeting to this association of distinguished workers in one of the most important fields of science. We desire to put at your service every facility we have that will add to your comfort and the success of your meeting. These recurring gatherings add to our obligations; for we feel that your work is a large contribution to every institution of learning, and because of our fortunate location we receive a direct and special benefit from your discussions. I should esteem it a great pleasure to listen to all of the able papers and remarks offered at this meeting.

In view of your annual gatherings here, it may not be out of place for me to say that the University is looking toward a larger life, better buildings, and equipment. We have secured a site on Seventeenth street, south of the State Department, and fronting on the President's park. This is in the immediate vicinity of the Agricultural Department, with its fine equipment of men and laboratories, which is working out great problems of public interest, and may also become helpful to many graduate students and men engaged in original research. In this location we hope to erect a well-equipped chemical laboratory and conduct graduate work of a high order. In this advanced work we shall desire to avail ourselves of the assistance of many scientists who are developing knowledge along these lines. We also hope that these buildings and laboratories, when erected, may furnish a temporary home and workshop for investigators who come to Washington for a longer or shorter period to avail themselves of the special advantages for research work existing in this city. For general assemblies like the present, we shall have better accommodations to offer, and they will be quite as convenient in all respects as the present location. It will take a little time to work out these plans and complete these buildings, and in the meantime the present building and equipment are yours while you are in the city and can use them.

There is one thought in my mind in reference to your work that I wish I might clearly express, and that is, the intellectual and spiritual benefit which is coming to mankind as a result of your labors. In the wonderful discoveries of science we are apt to think only of the immediate discovery and overlook the greater results that flow from it. You chemists are at the heart of things. You are discovering facts and laws which, in their natural operation, have far-reaching effects upon human life. The theory of evolution has fought its way and has been accepted. All recognize to-day that the things that are have come out from the past, formed and adapted by the environment in which the life found itself. The natural law of adaptation has worked and is working out the sublime objects of its author and human life has risen to higher and higher planes and to greater usefulness. But in these later years another thought has come to the world's students. If the environment has such an effect upon life, then why not change the environment and make it better; accepting the natural law the scientists are working to improve the conditions of life, and in this way are producing and will in the future produce a higher and a better life for man. In mechanics, after the machine, comes the search after more favorable conditions—the reduction of friction in order to secure better and greater results. So, having discovered a great law of life, the aim of the scientists is to improve the conditions, to relieve all unnecessary strain upon vital forces in order that the intellectual and spiritual life of man may have greater freedom and power of action. The man who finds himself upon a poor and unproductive soil need not take a long journey to new fields, but through the discoveries of chemistry he may make his land productive. Not only that, but he may know the products best adapted to the soil and climate in which he lives. Does the manufacturer find his profits diminishing and his capital threatened by the expense or the difficulties of production, the chemist will tell him how to increase the quality and decrease the expense. In almost every line of human activity where physical facts and forces are employed the chemist has become an indispensable man; and thus he is adding to the wealth of the nation and bringing within the reach of many, at a reasonable cost, the things which supply the ever increasing wants of our higher civilization.

Not only is the environment being changed for the better but by chemical investigations and experiments the foods we eat and the drinks we drink are purified, and their adaptation to our varying wants is made known. Thus the body is protected against harmful influences and relieved from much of the strain on the vital forces caused by the effort to overcome the bad effects of injurious matter; and by this selection, following the natural law, the best growth and development of the physical nature is secured. Thus is the work of the chemist making the conditions of physical life more favorable, and thereby he is making it possible to develop to a higher degree the intellectual and spiritual life. This result must always be regarded as the final goal of human life, and everything that tends to such development and enlargement is a noble work.

In the early history of this country the continent was a wilderness, but by the intelligent energy of our people, our frontiers have been driven back upon the undiscovered country and the wilderness has given up to civilized man its vast resources. So in the world of knowledge the great body of natural laws and facts was unknown until the scientists, by persistent effort and struggle, had driven back the boundary line of ignorance, and large fields in the realm of truth had been uncovered.

In the discovery of natural forces and laws and the rearrangement of matter to produce new and better conditions, man is working for the rejuvenation of man. Not by dreaming, nor by superstition, nor by the works of superstition, but by the direct, persistent, sincere, and fearless work of the student in the realms of nature is discovered that light which "shineth in darkness;" and then is brought into the great treasure house of humanity the facts and knowledge which are indeed "both new and old."

I congratulate you upon the splendid work you are doing, and trust that there will be many returns of the meeting of this association in the halls of this university.

The PRESIDENT. I am sure I express the thought of the association when I extend to President Needham our thanks for the words he has spoken. We appreciate what he has said, especially coming as it does from a man of his attainments and position.

We now come to the recommendations made by the referee on sugar. As he proposed, the recommendations made by the referee of last year will be referred to the committee having sugar under consideration for final adoption or other action. Are there any other papers on sugar?

Mr. SAWYER. I wish to submit a brief paper on methods of molasses analysis.

METHODS FOR THE ANALYSIS OF MOLASSES.

By H. E. SAWYER.

In examining several hundred samples of molasses during the last two years, I have found it impossible to obtain sufficiently accurate results by use of the analytical methods given in Bulletins Nos. 46 and 65. For the analysis of goods of fairly high quality, containing little coloring matter or other organic non-sugar, these methods are quite satisfactory; and for goods of lower grade they still are satisfactory, provided that approximate results are sufficient. But for the accurate analyses, such as ought to precede the acceptance of large lots of the low-grade molasses commonly worked up into alcohol, they are entirely inadequate.

The commercial chemists of our cities, on whose tests this molasses is usually bought and sold, have proceeded, in consequence of the lack of a proper official method, to follow each his own will and way in making analyses of these goods, with the result that satisfactory agreement between two analysts is very rare. In case of disagreement, who is to decide which chemist is in the right? And in case of serious disagreement and reference to the courts, how can an equitable decision be given in the absence of a reliable and official method? In consequence of this state of affairs, it is desirable for large buyers of molasses that the Association of Official Agricultural Chemists come to their assistance, as it did to the aid of the tanners a few years since, in developing and recommending analytical methods of suitable accuracy.

As I already have indicated, the inadequacy of the present methods in testing low-grade molasses is due to the intense coloration of such goods. To clarify a very dark and thick molasses sufficiently for polarization without the use of bone black may require 50 or 60 cc of basic lead acetate if the Ventzke normal weight is dissolved and made up to a volume of 100 cc, as recommended on page 39 of Bulletin No. 46. The bulk of the lead precipitate so produced concentrates the sugar solution and raises the polarization unduly. Moreover, the excess of lead remaining in the solution acts on the specific rotary power of the sugars and raises the polarization still further. I have known extreme cases where the direct polarization obtained with solutions so prepared was about a third in excess of what I believed to be the true value. That is to say, a reading of 22° was obtained when a value of 17° or 18° would have been nearer correct. In addition to its effect upon the direct polarization, the presence of a large excess of lead interferes with the inversion of a sugar solution and with the correct determination of the invert polarization and the sucrose.

To avoid difficulties of this sort, experiments were made during the last two years in the direction of using more dilute solutions than are recommended in the association's present methods. I dissolved the normal weight and made up to 500 cc; with this lower concentration I succeeded in getting filtrates light enough for polarization, using no more than 10–15 cc of basic acetate. The decrease in the bulk of the precipitate and in its influence upon the concentration of the solution, and the absence of the troublesome excess of lead from the solution, gave results nearer to the true polarization values and facilitated inversion.

Of course the polariscope readings on the dilute solutions in tubes of regulation length must be multiplied by five in order to obtain the actual polarization values, and it may be objected that this involves the multiplication of observation error. In fact, however, when the sum of five consecutive readings is taken as indicating the actual polarization, the probable error is very slight. With the triple field saccharimeter, showing clearly a difference of 0.05°, it may be a not uncommon experience to have five such readings agree absolutely; and in extreme cases of variation the probable error is surely smaller than the certain error involved in working on the lines of the present method.

To illustrate the closeness with which a polarization may be determined in the dilute solution I give the readings obtained in one of many tests upon this point.

	Degrees.
I. Sucrose solution containing approximately 6.5 grams per 100 cc. Polarized in 200 mm tube (five readings taken)	25.00 24.95 25.05 25.00 25.05
Average	25.01

	Degrees.	Degrees.
II. Sucrose solution exactly one-fifth the concentration of that used in No. I. Polarized in 200 mm tube (two sets of five readings each)	5.00 4.95 5.00 4.95 5.05	5.00 4.95 5.00 5.05 5.00
Sums	24.95	25.00
Mean errors of sums	±.09	±.075

It must be recognized by everyone who has had experience in molasses analysis that these errors are much smaller than those which attend the attempt to polarize the dark solutions prepared at the concentration of 26 grams to 100 cc.

It may be asked whether the polariscopic readings may not be materially affected by the change in the specific rotary powers of the sugars of molasses, due to the lowering of the concentration from 26 grams to 100 cc to 26 grams to 500 cc. I have found in practice that no effect of this sort is noticeable, and have calculated and here present the theoretical effect of such a change of concentration for a mixture of pure sucrose and invert sugar.

It may be assumed for the purpose of this calculation that the sirup contains 26.923 per cent of sucrose and an equal amount of invert sugar. These proportions are practically the same as those of low-grade molasses, and they give, according as the normal weight of 26 grams is dissolved to 100 or to 500 cc, concentrations of 7 or

1.40 grams per 100 cc for both sugars. The specific rotary powers and the corresponding Ventzke polarizations for the two concentrations are as follows:[a]

Polarizations with varying concentrations.

Concentration.		Sucrose.		Invert sugar.		Resultant direct polarization.
Sirup.	Sugar.	Specific rotation.	Polarization.	Specific rotation.	Polarization.	
		Angular degrees.	*Ventzke degrees.*	*Angular degrees.*	*Ventzke degrees.*	*Ventzke degrees.*
26:100	7.00:100	66.484	26.917	−19.910	−8.060	18.857
26:500	1.40:100	66.446	26.901	−19.708	−7.979	18.922
Difference.		....	.016	....	— .081	.065

Here, therefore, as in the observation, the possible error is materially smaller than the certain inaccuracies which attend the present method of molasses analysis.

Of course I do not claim that it is possible by using the dilute solution to determine with absolute accuracy the sucrose of a dark molasses. It is believed, however, that it is possible to insure increased accuracy, and that the method as outlined is a safe one, though still capable of further development. With this belief, and in the interest of the large molasses users I ask for it the consideration of the association.

Mr. **Munson**. I have no report to make, but as associate referee on sugar wish to say a few words on the work done during the past year. As stated at the last meeting, this work has for its purpose the obtaining of a more satisfactory alkaline tartrate solution, the idea being to get a solution that could be applied to all sugars. Considerable work has been done along this line during the past year, but at the present time there is nothing definite to report except that I have succeeded in getting a solution that has very much less effect on cane sugar than any of the solutions in use, even less than the Soxhlet solution, which is the most dilute and has the least effect on cane sugar of any solution now recognized. This solution requires 50 grams of caustic soda and 173 grams of rochelle salts per 500 cc. I have reduced the caustic soda to 24 grams per 500 cc and increased the rochelle salts to 275 grams. When working with a 20 per cent solution of pure cane sugar and using the Soxhlet method the reduction amounts to 50 mg of cuprous oxid, while with the new solution the reduction amounts to something less than 17 mg, about one-third of the effect so far as the action on cane sugar is concerned. With a 10 per cent solution of cane sugar, using the Soxhlet method, the reduction amounts to about 40 mg, while with the new solution the reduction is less than 11 mg. The idea has been to get a weak solution, one that will not reduce spontaneously and yet will have the least effect on cane sugar. It has been somewhat difficult to work this out. I think it would be a good idea to continue this work another year, and if we get a satisfactory solution for all the sugars, then to work out factors and a method for the various reducing sugars, using a given method and a given solution.

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[a] Optical Rotation of Organic Substances. Landolt-Long. 1902. Sucrose, p. 465; invert sugar, p. 592.

As the referee reported, there has been a committee appointed by the International Congress of Sugar Chemists, for the purpose of looking into this matter of a satisfactory solution for the reducing-sugar method, and I think it would be well for this association to authorize some cooperation.

The PRESIDENT. Is there any discussion.

Mr. WILEY. Some of our members may not know what the principal things are that the international committee has done. In the first place, it has recommended that the temperature at which all apparatus is standardized should be changed from 17.5° C to 20° C, as it is recognized that the former is too low. I was in favor of 22.5°, which is still 2° below the average temperature of our laboratories during the summer. We have made some interesting observations on this point, having kept the temperatures of polarization at the ports of New York and Boston every day for two years and have found the average to be over 23° C. It is seen that the adopted temperature is still far below our standard, but it is much better than before.

The next most important resolution of the international committee is that all polarizations shall be made at the temperature at which the instruments are graduated, but each country is allowed to have its instruments graduated at its own standard temperature if it be higher or lower than 20°. Thus the errors due to graduating instruments at one temperature and making polarizations at another are eliminated. I think that is the most important work of the international committee.

The third is that a true cubic centimeter flask shall be used with 26 grams of sugar instead of 26.048 grams with the Mohr flask. That eliminates an element of discord which never should have entered into sugar analysis at all.

These are the most important resolutions of the committee. It is working all the time and is now preparing one standard solution of sugar. Thus this principle, which started with the organization of this association, has permeated all the world, and instead of using instruments standardized by everyone who uses them, there is one source from which standard sugars can be obtained, that is, from the president of the international committee. Each country is allowed to make its own standard sugar if it wants to, but it is far better to get it from one source. We have received a package for the United States, a part of which has been given to the National Bureau of Standards. The members of this association ought to know that the National Bureau of Standards will standardize instruments free of cost for all Government institutions, and I think that the agricultural experiment stations come under that category. It is most important in sugar analysis to know that the polariscope is right. While formerly the differences in polarization often averaged as much as 0.8 per cent the comparative data to-day rarely differ more than 0.1 per cent, which shows what has been accomplished by the adoption of these uniform methods.

The **President**. I understand that the committee on food standards is ready to report.

REPORT OF THE COMMITTEE ON FOOD STANDARDS.

The President and Members of the Association of Official Agricultural Chemists: On behalf of the standing committee on food standards, I desire to report that Congress, by an act approved March 3, 1903, made further provision "to enable the Secretary of Agriculture in collaboration with the Association of Official Agricultural Chemists, and such other experts as he may deem necessary, to establish standards of purity for food products and to determine what are regarded as adulterations therein, for the guidance of the officials of the various States and the courts of justice."

Acting under commission from the Secretary of Agriculture and the authority conferred by this association, the committee has, during the past year, prepared a further revision of several of the schedules to which your attention and that of trade and commercial interests concerned have already been called, and has conducted a voluminous correspondence respecting the definitions and standards proposed.

Three meetings have been held, of an aggregate duration of sixteen days, during which numerous hearings have been given to trade interests requesting an opportunity for conference and for discussion of the definitions and standards. It is a pleasure to report that, while naturally solicitous lest hasty and ill-considered action might needlessly disturb the vast business interests concerned, the representatives of those engaged in food manufacture and commerce have expressed a cordial approval of the movement as conducive to the welfare of both producer and consumer.

Your committee recommends to the Secretary of Agriculture for approval at this time standards for certain articles belonging to the meat, milk, sugar, condiment, and cocoa schedules. They are hereto appended:

Before the adoption of any schedule, it has been submitted to the manufacturing firms and the trade immediately interested for criticism and, when requested by them, conferences for discussion have been arranged. Certain questions have arisen in the discussion of these standards relative to several substances sometimes used as preservatives or coloring matters; in the judgment of the committee these questions can most satisfactorily be treated in connection with Schedule III, Preservatives and coloring matters, and recommendations have therefore been deferred pending the consideration of that schedule.

For the primary definitions and standards and for the compilations of data for standards and constant assistance in the revision of the schedules, the committee is greatly indebted to the following persons:

Charles D. Woods, Ph.D., director of the Maine Agricultural Experiment Station, Orono, Me., referee on meat and its products; L. L. Van Slyke, Ph.D., chemist of the New York Agricultural Experiment Station, Geneva, N. Y., referee on milk and its products; Charles A. Crampton, M. D., chemist of the Bureau of Internal Revenue, referee on beverages, including cocoa and cocoa products; A. L. Winton, Ph.B., chemist of the Connecticut Agricultural Experiment Station, New Haven, Conn., referee on condiments.

The committee is also indebted to others for information and helpful suggestions, which will be more specifically acknowledged in a report of its work to be later submitted.

Very respectfully,

William Frear.
E. H. Jenkins.
M. A. Scovell.
H. A. Weber.
H. W. Wiley.

Standards of Purity for Food Products

Whereas the Congress of the United States by an act approved June 3, 1902, authorized the Secretary of Agriculture to establish standards of purity for food products; and

Whereas he was empowered by this act to consult with the committee on food standards of the Association of Official Agricultural Chemists and other experts in determining these standards; and

Whereas he has in accordance with the provisions of the act availed himself of the counsel and advice of these experts and of the trade interests touching the products for which standards have been determined, and has reached certain conclusions based on the general principles of examination and conduct hereinafter mentioned;

Therefore, I, James Wilson, Secretary of Agriculture, do hereby proclaim and establish the following standards for purity of food products together with their precedent definitions as the official standards of these food products for the United States of America.

James Wilson.

Washington, D. C., *November 21, 1903.*

PRINCIPLES ON WHICH THE DEFINITIONS AND STANDARDS ARE BASED.

The general considerations which have guided the committee in preparing the definitions and standards for food products are the following:

1. The main classes of food articles are defined before the subordinate classes are considered.
2. The names of the various substances for which standards are proposed are defined.
3. The definitions are so framed as to exclude from the articles defined substances not included in the definitions.
4. The definitions include, where possible, those qualities which make the articles described wholesome for human food.
5. A term defined in any of the several schedules has the same meaning wherever else it is used in this report.
6. The names of food products herein defined usually agree with existing American trade or manufacturing usage, but where such usage is not clearly established or where trade names confuse two or more articles for which specific designations are desirable, preference is given to one of the several trade names applied.
7. Standards are based upon data representing materials produced under American conditions and manufactured by American processes or representing such varieties of foreign articles as are chiefly imported for American use.
8. The standards fixed are such that a departure of the articles to which they apply, above the maximum or below the minimum limit prescribed is evidence that such articles are of inferior or abnormal quality.
9. The limits fixed as standard are not necessarily the extremes authentically recorded for the article in question, because such extremes are commonly due to abnormal conditions of production and are usually accompanied by marks of inferiority or abnormality readily perceived by the producer or manufacturer.

FOOD DEFINITIONS AND STANDARDS.

I. ANIMAL PRODUCTS.

A. MEATS AND THE PRINCIPAL MEAT PRODUCTS.

(a) MEATS.

Definitions.

1. *Meat* is any sound, dressed, and properly prepared edible part of animals in good health at the time of slaughter. The term "animals," as herein used, includes not only mammals, but fish, fowl, crustaceans, mollusks, and all other animals used as food.

2. *Fresh meat* is meat from animals recently slaughtered or preserved only by refrigeration.

3. *Salted*, *pickled*, and *smoked meats* are unmixed meats preserved by salt, sugar, vinegar, spices, or smoke, singly or in combination, whether in bulk or in packages.

Standard.

Standard meat, fresh meat, and salted, pickled, and smoked meats are such as conform respectively to the foregoing definitions.

(b) MANUFACTURED MEATS.

Definition.

1. *Manufactured meats* are meats not included in definitions 2 and 3, whether simple or mixed, whole or comminuted, in bulk or packages, with or without the addition of salt, sugar, vinegar, spices, smoke, oils, or rendered fat.

Standard.

Standard manufactured meats conform to the foregoing definition. If they bear names descriptive of composition, they correspond thereto, and when bearing such descriptive names, if force or flavoring meats are used, the kind and quantity thereof are made known.

(c) MEAT EXTRACTS, MEAT PEPTONES, ETC.

(Schedule in preparation.)

(d) LARD.

Definitions.

1. *Lard* is the rendered fresh fat from slaughtered, healthy hogs.

2. *Leaf lard* is the lard rendered at moderately high temperatures from the internal fat of the abdomen of the hog, excluding that adherent to the intestines.

Standard.

Standard lard and standard leaf lard are lard and leaf lard, respectively, free from rancidity, containing not more than one (1) per cent of substances, other than fatty acids, not fat, necessarily incorporated therewith in the process of rendering, and standard leaf lard has an iodine number not greater than sixty (60).

Definition.

3. *Neutral lard* is lard rendered at low temperatures.

B. MILK AND ITS PRODUCTS.

(a) MILKS.

Definition.

1. *Milk* (*whole milk*) is the lacteal secretion obtained by the complete milking of one or more healthy cows, properly fed and kept, excluding that obtained within fifteen days before and five days after calving.

Standard.

Standard milk is milk containing not less than twelve (12) per cent of total solids and not less than eight and one-half (8.5) per cent of solids not fat, nor less than three and one-fourth (3.25) per cent of milk fat.

Definitions.

2. *Blended milk* is milk modified in its composition so as to have a definite and stated percentage of one or more of its constituents.
3. *Skim milk* is milk from which a part or all of the cream has been removed.

Standard.

Standard skim milk is skim milk containing not less than nine and one-fourth (9.25) per cent of milk solids.

Definitions.

4. *Buttermilk* is the product that remains when butter is removed from milk or cream in the process of churning.
5. *Pasteurized milk* is standard milk that has been heated below boiling but sufficiently to kill most of the active organisms present and immediately cooled to fifty degrees (50°) Fahr. or lower to retard the development of their spores.
6. *Sterilized milk* is standard milk that has been heated at the temperature of boiling water or higher for a length of time sufficient to kill all organisms present.
7. *Condensed milk* is milk from which a considerable portion of water has been evaporated.
8. *Sweetened condensed milk* is milk from which a considerable portion of water has been evaporated and to which sugar (sucrose) has been added.

Standard.

Standard condensed milk and *standard sweetened condensed milk* are condensed milk and sweetened condensed milk, respectively, containing not less than twenty-eight (28) per cent of milk solids, of which not less than one-fourth is milk fat.

Definition.

9. *Condensed skim milk* is skim milk from which a considerable portion of water has been evaporated.

(*b*) MILK FAT OR BUTTER FAT

Definition.

1. *Milk fat* or *butter fat* is the fat of milk.

Standard.

Standard milk fat or butter fat has a Reichert-Meissl number not less than twenty-four (24) and a specific gravity not less than 0.905 (40° C. /40° C.).

(*c*) CREAM.

Definition.

1. *Cream* is that portion of milk, rich in butter fat, which rises to the surface of milk on standing, or is separated from it by centrifugal force.

Standard.

Standard cream is cream containing not less than eighteen (18) per cent of milk fat.

Definition.

2. *Evaporated cream* is cream from which a considerable portion of water has been evaporated.

(*d*) BUTTER.

Definition.

1. *Butter* is the product obtained by gathering in any manner the fat of fresh or ripened milk or cream into a mass, which also contains a small portion of the other milk constituents, with or without salt. By acts of Congress approved August 2, 1886, and May 9, 1902, butter may also contain additional coloring matter.

Standard.

Standard butter is butter containing not less than eighty-two and five-tenths (82.5) per cent of butter fat.

Definition.

2. *Renovated or process butter* is the product obtained by melting butter and reworking, without the addition or use of chemicals or any substances except milk, cream, or salt.

Standard.

Standard renovated or process butter is renovated or process butter containing not more than sixteen (16) per cent of water and at least eighty-two and five-tenths (82.5) per cent of butter fat.

(*e*) CHEESE.

Definitions.

1. *Cheese* is the solid and ripened product obtained by coagulating the casein of milk by means of rennet or acids, with or without the addition of ripening ferments and seasoning. By act of Congress, approved June 6, 1896, cheese may also contain additional coloring matter.

2. *Whole-milk or full-cream cheese* is cheese made from milk from which no portion of the fat has been removed.

3. *Skim-milk cheese* is cheese made from milk from which any portion of the fat has been removed.

4. *Cream cheese* is cheese made from milk and cream, or milk containing not less than six (6) per cent of fat.

Standard.

Standard whole-milk cheese or full-cream cheese is whole-milk or full-cream cheese containing in the water-free substance not less than fifty (50) per cent of butter fat.

(*f*) MISCELLANEOUS MILK PRODUCTS.

Definition.

1. *Ice cream* (in preparation).

Standard.

Standard ice cream (in preparation).

Definitions.

2. *Whey* is the product remaining after the removal of fat and casein from milk in the process of cheese making.

3. *Kumiss* is mare's or cow's milk, with or without the addition of sugar (sucrose), which has undergone alcoholic fermentation.

II. VEGETABLE PRODUCTS.

A. GRAIN PRODUCTS.

(Schedule in preparation.)

B. FRUITS AND VEGETABLES.

(Schedule in preparation.)

C. SUGARS AND RELATED SUBSTANCES.

(a) SUGAR AND SUGAR PRODUCTS.

Definition.

1. *Sugar* is the product chemically known as sucrose (saccharose), chiefly obtained from sugar cane, sugar beets, sorghum, maple, or palm.

Standard.

Standard sugar is white sugar containing at least ninety-nine and five-tenths (99.5) per cent of sucrose.

Definitions.

2. *Granulated, loaf, cut, milled, and powdered sugars* are different forms of standard sugars.
3. *Maple sugar* is the solid product resulting from the evaporation of maple sap.
4. *Massecuite, melada, mush sugar, and concrete* are products obtained by evaporating the purified juice of a sugar-producing plant, or a solution of sugar, to a solid or semi-solid consistence in which the sugar chiefly exists in a crystalline state.
5. *Molasses* is the product left after separating the sugar from massecuite, melada, mush sugar, or concrete.

Standard.

Standard molasses is molasses containing not more than twenty-five (25) per cent of water nor more than five (5) per cent of ash.

Definitions.

6. *Sirup* is the product obtained by purifying and evaporating the juice of a sugar-producing plant without removing any of the sugar.
7. *Sugar-cane sirup* is a sirup obtained by the evaporation of the juice of the sugar cane or by the solution of sugar-cane concrete.
8. *Sorghum sirup* is a sirup obtained by the evaporation of sorghum juice or by the solution of sorghum concrete.
9. *Maple sirup* is a sirup obtained by the evaporation of maple sap or by the solution of maple concrete.
10. *Sugar sirup* is a product obtained by dissolving sugar to the consistence of a sirup.

Standard.

Standard sirup is a sirup containing not more than thirty (30) per cent of water nor more than two and five-tenths (2.5) per cent of ash.

(b) GLUCOSE PRODUCTS.

Definition.

1. *Starch sugar or grape sugar* is the solid product obtained by hydrolyzing starch or a starch-containing substance until the greater part of the starch is converted into dextrose. Starch sugar or grape sugar appears in commerce in two forms, anhydrous and hydrous. In the former, the sugar is crystallized without water of crystallization; in the latter, it is crystallized with water of crystallization. The hydrous varieties are commonly known as 70 and 80 sugars; 70 sugar is also known as brewers' sugar, and 80 sugar as climax or acme sugar.

Standards.

(a) *Standard 70 sugar or brewers' sugar* is hydrous starch sugar containing not less than seventy (70) per cent of dextrose and not more than eight-tenths (0.8) per cent of ash.

(b) *Standard 80 sugar, climax or acme sugar,* is hydrous starch sugar containing not less than eighty (80) per cent of dextrose and not more than one and one-half (1.5) per cent of ash.

(c) *Standard anhydrous grape sugar* is anhydrous grape sugar containing not less than ninety-five (95) per cent of dextrose without water of crystallization and not more than eight-tenths (0.8) per cent of ash.

The ash of these standard products consists almost entirely of chlorids and sulphates of lime and soda.

Definition.

2. *Glucose, mixing glucose, or confectioners' glucose* is a thick sirupy substance obtained by incompletely hydrolizing starch or a starch-containing substance, decolorizing and evaporating the product. It is found in various degrees of concentration, ranging from forty-one (41) to forty-five (45) degrees Baumé.

Standard.

Standard glucose, mixing glucose, or confectioners' glucose is colorless glucose, varying in density between forty-one (41) and forty-five (45) degrees Baumé, at a temperature of one hundred (100) degrees F. (37.7° C.). It conforms in density, within these limits, to the degree Baumé it is claimed to show, and for a density of forty-one (41) degrees Baumé contains not more than twenty-one (21) per cent of water and for a density of forty-five (45) degrees not more than fourteen (14) per cent. It contains on a basis of forty-one (41) degrees Baumé not more than one (1) per cent of ash, consisting chiefly of chlorids and sulphates of lime and soda.

Definition.

3. *Glucose sirup or corn sirup* is glucose unmixed or mixed with sirup or molasses.

Standard.

Standard glucose sirup or *corn sirup* is glucose sirup or corn sirup containing not more than twenty-five (25) per cent of water nor more than three (3) per cent of ash.

(*c*) CANDY.

Definition.

1. *Candy* is a product prepared from a saccharine substance or substances, with or without the addition of harmless coloring, flavoring, or filling materials.

Standard.

Standard candy is candy containing no terra alba, barytes, talc, chrome yellow, or other mineral substances or poisonous colors or flavors or other ingredients injurious to health.

(*d*) HONEY.

(Schedule in preparation.)

D. CONDIMENTS (EXCEPT VINEGAR).

(*a*) SPICES.

General Definition.

1. *Spices* are aromatic vegetable substances used for the seasoning of food.

General Standard.

Standard spices are sound spices, true to name, from which no portion of any volatile oil or other flavoring principle has been removed.

Definition.

2. *Allspice or pimento* is the dried fruit of *Pimenta officinalis* Lindl.

Standard.

Standard allspice is allspice containing not less than eight (8) per cent of quercitannic acid;[a] not more than six (6) per cent of total ash; not more than five-tenths (0.5) per cent of ash insoluble in hydrochloric acid, and not more than twenty-five (25) per cent of crude fiber.

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[a] Calculated from the total oxygen absorbed by the aqueous extract.

Definitions.

3. *Anise* is the fruit of *Pimpinella anisum* L.
4. *Bay leaf* is the dried leaves of *Laurus nobilis* L.
5. *Capers* are the flower buds of *Capparis spinosa* L.
6. *Caraway* is the fruit of *Carum carvi* L.

CAYENNE AND RED PEPPERS.

7. *Red pepper* is the red, dried, ripe fruit of any species of *Capsicum*.
8. *Cayenne pepper or cayenne* is the dried, ripe fruit of *Capsicum fastigiatum* DC., *Capsicum frutescens* L., *Capsicum baccatum* L., or some other small-fruited species of *Capsicum*.

Standard.

Standard cayenne pepper is cayenne pepper containing not less than fifteen (15) per cent of nonvolatile ether extract; not more than six and five-tenths (6.5) per cent of total ash; not more than five-tenths (0.5) per cent of ash insoluble in hydrochloric acid; not more than one and five-tenths (1.5) per cent of starch by the diastase method, and not more than twenty-eight (28) per cent of crude fiber.

Definitions.

9. *Celery seed* is the dried seed of *Apium graveolens* L.
10. *Cinnamon* is the dried bark of any species of the genus *Cinnamomum* from which the outer layers may or may not have been removed.
11. *True cinnamon* is the dried inner bark of *Cinnamomum zeylanicum* Breyne.
12. *Cassia* is the dried bark of various species of *Cinnamomum*, other than *Cinnamomum zeylanicum*, from which the outer layers may or may not have been removed.
13. *Cassia buds* are the dried immature fruit of species of *Cinnamomum*.
14. *Ground cinnamon or ground cassia* is a powder consisting of cinnamon, cassia, or cassia buds, or a mixture of these spices.

Standard.

Standard cinnamon or cassia is cinnamon or cassia containing not more than eight (8) per cent of total ash and not more than two (2) per cent of sand.

Definition.

15. *Cloves* are the dried flower buds of *Eugenia caryophyllata* Thunb. (*Caryophyllus aromaticus* L.) which contain not more than five (5) per cent of clove stems.

Standard.

Standard cloves are cloves containing not less than ten (10) per cent of volatile ether extract; not less than twelve (12) per cent of quercitannic acid;[a] not more than eight (8) per cent of total ash; not more than five-tenths (0.5) per cent of ash insoluble in hydrochloric acid, and not more than ten (10) per cent of crude fiber.

Definitions.

16. *Coriander* is the dried fruit of *Coriandrum sativum* L.
17. *Cumin seed* is the fruit of *Cuminum cyminum* L.
18. *Dill seed* is the fruit of *Peucedanum graveolens* Benth. & Hook.
19. *Fennel* is the fruit of *Foeniculum vulgare* Gaertn.
20. *Ginger* is the washed and dried, or decorticated and dried, rhizome of *Zingiber officinale* Roscoe.

Standard.

Standard ginger is ground or whole ginger containing not less than forty-two (42) per cent of starch by the diastase method nor less than forty-six (46) per cent by direct inversion,[b] not more than eight (8) per cent of crude fiber, not more than eight (8) per cent of total ash, not more than one (1) per cent of lime, and not more than three (3) per cent of ash insoluble in hydrochloric acid.

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[a] Calculated from the total oxygen absorbed by the aqueous extract.

[b] Copper-reducing matters by direct inversion calculated as starch.

Definition.

21. *Limed or bleached ginger* is whole ginger coated with carbonate of lime.

Standard.

Standard limed or bleached ginger is limed or bleached ginger containing not more than ten (10) per cent of ash, not more than four (4) per cent of carbonate of lime, and conforming in other respects to standard ginger.

Definition.

22. *Horse-radish* is the root of *Cochlearia armoracia* L.

Standard.

Standard grated or ground horse-radish may be mixed with vinegar.

Definition.

23. *Mace* is the dried arillus of *Myristica fragrans* Houttuyn.

Standard.

Standard mace is mace containing not less than twenty (20) nor more than thirty (30) per cent of nonvolatile ether extract, not more than three (3) per cent of total ash, not more than five-tenths (0.5) per cent of ash insoluble in hydrochloric acid, and not more than ten (10) per cent of crude fiber.

Definitions.

24. *Macassar or Papua mace* is the dried arillus of *Myristica argentea* Warb.
25. *Bombay mace* is the dried arillus of *Myristica malabarica* Lamarck.
26. *Marjoram* is the leaves, flowers, and branches of *Origanum majorana* L.
27. *Mustard seed* is the seed of *Sinapis alba* L. (white mustard), *Brassica nigra* Koch (black mustard), or *Brassica juncea* Coss. (black or brown mustard).
28. *Ground mustard* is a powder made from mustard seed, with or without the removal of the hulls and a portion of the fixed oil.

Standard.

Standard ground mustard is mustard containing not more than two and five-tenths (2.5) per cent of starch by the diastase method and not more than eight (8) per cent of total ash.

Definition.

29. *Nutmeg* is the dried seed of *Myristica fragrans* Houttuyn, deprived of its testa and with or without a thin coating of lime.

Standard.

Standard nutmegs, ground or unground, are nutmegs containing not less than twenty-five (25) per cent of nonvolatile ether extract; not more than five (5) per cent of total ash; not more than five-tenths (0.5) per cent of ash insoluble in hydrochloric acid, and not more than ten (10) per cent of crude fiber.

Definitions.

30. *Macassar, Papua, male, or long nutmeg* is the dried seed of *Myristica argentea* Warb., deprived of its testa.
31. *Paprica* is the dried ripe fruit of *Capsicum annuum* L., *Capsicum longum* DC., or some other large-fruited species of *Capsicum*.

PEPPER.

32. *Black pepper* is the dried immature berries of *Piper nigrum* L.

Standard.

Standard black pepper is black pepper free from added pepper shells, pepper dust, and other pepper by-products, and containing not less than six (6) per cent of nonvolatile ether extract; not less than twenty-two (22) per cent of starch by the diastase method; not less than twenty-eight (28) per cent of starch by direct inversion;[a] not more than seven (7) per cent of total ash; not more than two (2) per cent of ash insoluble in hydrochloric acid, and not more than fifteen (15) per cent of crude fiber. One hundred parts of the nonvolatile ether extract contain not less than three and one-fourth (3.25) parts of nitrogen.

Definitions.

33. *Long pepper* is the dried fruit of *Piper longum* L.

34. *White pepper* is the dried mature berries of *Piper nigrum* L., from which the outer coating, or the outer and inner coatings, have been removed.

Standard.

Standard white pepper is white pepper containing not less than six (6) per cent of nonvolatile ether extract; not less than fifty-three (53) per cent of starch by the diastase method; not less than forty (40) per cent of starch by direct inversion;[a] not more than four (4) per cent of total ash; not more than five-tenths (0.5) per cent of ash insoluble in hydrochloric acid, and not more than five (5) per cent of crude fiber. One hundred parts of the nonvolatile ether extract contain not less than four (4) parts of nitrogen.

Definitions.

35. *Saffron* is the dried stigmas of *Crocus sativus* L.

36. *Sage* is the leaves of *Salvia officinalis* L.

37. *Savory, or summer savory,* is the leaves, blossoms, and branches of *Satureia hortensis* L.

38. *Thyme* is the leaves and ends of blooming branches of *Thymus vulgaris* L.

(*b*) FRUIT EXTRACTS.

(Schedule in preparation.)

(*c*) SALAD OILS.

(Schedule in preparation.)

(*d*) SALT.

(Schedule in preparation.)

E. BEVERAGES (AND VINEGAR).

(*a*) TEA.

(Schedule in preparation.)

(*b*) COFFEE.

(Schedule in preparation.)

(*c*) COCOA AND COCOA PRODUCTS.

Definitions.

1. *Cocoa beans* are the seeds of the cacao tree, *Theobroma cacao* L.

2. *Cocoa nibs, or cracked cocoa* is the roasted, broken cocoa bean freed from its shell or husk.

3. *Chocolate, plain or bitter, chocolate liquor,* is the solid or plastic mass obtained by grinding cocoa nibs without the removal of fat or other constituents except the germ.

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[a] Copper-reducing matters by direct inversion calculated as starch.

Standard.

Standard chocolate is chocolate containing not more than three (3) per cent of ash insoluble in water, three and fifty-hundredths (3.50) per cent of crude fiber and nine (9) per cent of starch nor less than forty-five (45) per cent of cocoa fat.

Definition.

4. *Sweet chocolate and chocolate coatings* are plain chocolate mixed with sugar (sucrose), with or without the addition of cocoa butter, spices, or other flavoring materials.

Standard.

Standard sweet chocolate and standard chocolate coating are sweet chocolate and chocolate coating containing in the sugar- and fat-free residue no higher percentage of either ash, fiber, or starch than is found in the sugar- and fat-free residue of plain chocolate.

Definition.

5. *Cocoa or powdered cocoa* is cocoa nibs, with or without the germ, deprived of a portion of its fat and finely pulverized.

Standard.

Standard cocoa is cocoa containing percentages of ash, crude fiber, and starch corresponding to those in chocolate after correction for fat removed.

Definition.

6. *Sweet or sweetened cocoa* is cocoa mixed with sugar (sucrose).

Standard.

Standard sweet cocoa is sweet cocoa containing not more than sixty (60) per cent of sugar (sucrose) and in the sugar- and fat-free residue no higher percentage of either ash, crude fiber, or starch than is found in the sugar- and fat-free residue of plain chocolate.

(*d*) FRUIT JUICES—FRESH, SWEET, AND FERMENTED.

(In preparation.)

(*e*) VINEGAR.

(In preparation.)

(*f*) MEAD, ROOT BEER, ETC.

(*g*) MALT LIQUORS.

(In preparation.)

(*h*) SPIRITUOUS LIQUORS.

(In preparation.)

(*i*) CARBONATED WATERS, ETC.

III. PRESERVATIVES AND COLORING MATTERS.

(In preparation.)

The PRESIDENT. What disposition will the association make of this report?

Mr. WILEY. As Congress has authorized the Secretary of Agriculture to proclaim and establish standards of purity for food products for the whole country and you have commissioned this committee to prepare them, it would seem to be entirely sufficient to receive the report and await the action of the Secretary. I might say that the Secretary of Agriculture has followed the work of the committee in its progress and is ready to affix his signature to the proclamation, now in the process of engrossing, which will make these the official standards of the United States.

Upon motion the report of the committee was received.

Mr. WILEY. We have with us a committee of the American Chemical Society on the purity of reagents, which is considering matters of great importance to this association. I move that Mr. Hillebrand, the chairman of this committee, be invited to make a statement in regard to this work.

ADDRESS OF W. F. HILLEBRAND, AMERICAN CHEMICAL SOCIETY.

I would make one correction in Mr. Wiley's statement. President Long is ex officio chairman of this committee and I, as secretary, have been delegated to present to you the object of this committee—what we desire to accomplish and what we ask of you.

The committee was appointed nearly a year ago at the meeting of the American Chemical Society in this city, having in view the extreme importance to chemists of obtaining a better quality of chemical reagents than is now generally to be had. It is unnecessary probably to dwell upon the importance of this matter to a body constituted as this one is. The committee has found it difficult by correspondence to obtain a fair exchange of views, and therefore a meeting was called to be held in this city and it has been in session during yesterday and this forenoon. The committee consists of President Long as chairman, with Messrs. Baskerville, Dennis, Talbot, and myself as fellow-members. With this committee by invitation sat Messrs. Noyes, chief chemist, and H. N. Stokes, chemist, of the National Bureau of Standards. There ensued at the start a lengthy discussion as to the manner in which the cooperation of the National Bureau of Standards might be made effective. The proposition which eventually met with favor was to the following effect:

A set of specifications and tests for chemicals in different grades having been prepared and distributed, it is hoped that chemists will generally make acceptance of their purchases conditional upon their coming up to the specifications as to purity set forth in the contract or order. Dealers or manufacturers will, it is hoped, make use of a special label, to be attached to the original package, which shall be a guaranty that the contents come up to the requirements of the published list for any particular grade. If the purchaser finds that the quality implied by the label has not been supplied, he may request the National Bureau of Standards to confirm or refute his judgment by an analysis made upon the contents of an unopened original package forming part of his purchase. The certificate of the Bureau of Standards will then be just ground for rejecting the shipment in case the quality is inferior to that guaranteed. The furnishing of reagents of extraordinary purity must be a matter for special agreement between the purchaser and dealer or manufacturer. The latter may, if he desires, have a large importation or supply otherwise obtained, sampled and bottled by an agent of the Bureau, who shall then take a sample or samples for official tests. If the results of the tests are satisfactory the bottles shall be provided with an official seal of the Bureau, bearing the date and something to indicate the particular grade of chemical as listed in the published document already alluded to. This list of tests and specifications shall be prepared, presumably, under the direction of this committee, and shall be issued only with the approval of and, if possible, under the auspices of the National Bureau of Standards. It is expected that a fee will be charged for examinations made by the Bureau.

On the basis of Krauch's book, Prüfung chemischer Reagentien, a provisional list of those chemicals which should be considered at the start has been prepared, it being the prevailing sentiment that the compilation of specifications and tests should not be a duplication of the above work, but should be one made independently with special regard to the needs of chemists in this country and that it should be prepared in less detail.

This is a very simple outline of our aims. We desire to improve the standard of chemicals furnished chemists in this country. We realize that this can not be brought about immediately. It is a work involving a great deal of labor and a great deal of correspondence, and one which demands the cooperation of chemists in all lines of chemical work throughout the country. The details of the scheme are, of course, yet to be worked out. The first step is to prepare a list of the specifications, tests, and chemicals that shall be included in this. It has been thought that at the start it would not be wise to reduplicate Krauch's large book with all the chemicals there listed and the details therein given; but to prepare a list which shall meet the principal needs of the chemists of this country. And therefore a provisional list of chemicals has been made out, which is subject to revision or addition, and this list has been subdivided among the members of the committee, who are requested to correspond with experts throughout the country and with dealers in this country and abroad to find out what grades of chemicals shall be specified and to what degree of purity each grade must conform. This is a work involving a great deal of labor and it will take several years to accomplish it entirely. It is a thing which must begin in a small way and gradually expand.

It is earnestly hoped that this association will appoint a committee to cooperate with the committee of the American Chemical Society, the Association of Official Agricultural Chemists' committee having as its work (1) the specification of those chemicals which are used particularly in its work; (2) the tests of these reagents in their different grades—if more than one grade is used; (3) the requirements that these reagents must meet in order to satisfy the demands; and (4) that your committee will report results of its deliberations to our committee, so that we may use your results in furthering the aims of this work. [Applause.]

The **President**. I feel sure that the association will do all it can to further this work and perhaps some action will be taken before our adjournment.

Proceeding with the order of business, the next report is that on ash. Mr. Fraps, the referee, is not present but his report will be presented by the secretary.

REPORT ON ASH.

By G. S. Fraps, *Referee.*

The referee accepted the position with some reluctance last spring, upon the resignation of Mr. E. G. Runyan, the original appointee, since he had served in this capacity twice before. Various changes during the year have interfered with the work and it is regretted that there is not more to report at this meeting.

The work for the year was confined to a study of methods for the determination of sulphur and sulphates. About seven chemists having signified their willingness to take part in the work, samples were prepared and sent out with the following directions:

Directions for Work.

The samples consist of cowpeas and cotton-seed meal for the total sulphur determination, and sorghum for the sulphate work. Mix each sample thoroughly before analysis. Make at least two determinations by each method. In reporting give weights of precipitates, per cent of sulphur (SO_3) on samples as received and on dry matter. Please report results as early as possible, and give the referee the benefit of your suggestions in regard to the work.

Compare the two following methods for total sulphur, using the cotton-seed meal and cowpeas:

SULPHUR.

Method I.—Place 5 grams material in a 2½-inch porcelain evaporating dish, add 20 cc concentrated nitric acid, and heat the mixture cautiously on the water bath until all danger of overflowing has passed. Then evaporate almost to dryness, add 10 cc of a 5 per cent solution of potassium nitrate, evaporate to complete dryness, and ignite, at first gently, then more vigorously, until the residue is white. The residue is then dissolved in hydrochloric acid and evaporated to dryness, and heated for some time in an air bath to render silica insoluble. Take up in water with the addition of a little hydrochloric acid, filter to 150 cc or more, precipitate the sulphuric acid with a solution of 1 gram barium chlorid, and complete the determination in the usual way. Make a blank test with the reagents.

Method II.—Fifteen grams of sodium peroxid are weighed and most of it introduced at once into a silver or nickel crucible and converted into hydroxid by adding a little water and boiling, preferably over an alcohol lamp, until the excess of water is expelled. The hydroxid is allowed to cool until pasty, when 2 grams of the material is stirred in it as quickly as possible. The heat is then raised and the remainder of the peroxid added in small portions to complete the oxidation. The fused mass, after cooling, is dissolved in water, neutralized with hydrochloric acid, evaporated to dryness, and heated in an air bath for some time to render silica insoluble. The residue is then taken up in water, with the addition of a little hydrochloric acid, filtered and washed to about 400 cc, and the sulphuric acid precipitated as before. Make a blank test with the reagents.

SULPHATES.

Determine the sulphates in the sample of sorghum by the following method:

Five grams of substance are mixed well with 50 cc of a 1 per cent solution of hydrochloric acid, allowed to stand half an hour, filtered, and washed with the dilute acid to 250 cc or more. The liquid is heated to boiling, barium chlorid added, and the determination completed in the usual way.

MOISTURE.

The results on moisture are stated as a matter of record.

TABLE I.—*Moisture determinations.*

Analyst.	No. 1. Cotton-seed meal.	No. 2. Cowpeas.	No. 3. Sorghum.
	Per cent.	*Per cent.*	*Per cent.*
F. P. Veitch, Washington, D. C	5.98	8.55	7.75
Frank T. Shutt, Ottawa, Canada	5.90	8.43	7.14
	6.00	8.32	7.21
R. W. Thatcher, Washington (State)	5.35	7.90	6.36
	5.20	7.74	6.20
W. G. Morrison, North Carolina	6.28	8.59	....
G. S. Fraps, Texas	5.89	8.28	....

REMARKS OF ANALYSTS.

R. W. Thatcher.—Moisture was determined by drying seven hours in a boiling-water oven. It is my practice to dry in hydrogen, but appliances for this work were not available at the time this work was done.

G. S. Fraps.—The moisture determinations were made by drying eight hours on a watch glass at 100° C. It is my observation that drying five hours in a current of hydrogen gives usually higher results than by drying on a watch glass.

TOTAL SULPHUR.

The percentages of sulphur obtained by the methods to be compared are presented in the following table:

TABLE II.—*Determination of sulphur as* SO_3.

Analyst.	Cowpeas.		Cotton-seed meal.	
	Nitric-acid method.	Peroxid method.	Nitric-acid method.	Peroxid method.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*
F. P. Veitch, Washington, D. C.	0.466	0.539	1.071	1.133
Frank T. Shutt, Ottawa, Canada	.490	....	1.115	....
	.500	....	1.098	....
R. W. Thatcher, Washington (State)	.371	....	.944	....
	.379	....	.953	....
W. G. Morrison, North Carolina	....	.583	....	1.170
	....	.593	....	1.013
G. S. Fraps, Texas	.524	.501	1.120	1.129
	.532	.515	....	1.230
Average of all determinations	.466	.544	1.048	1.135

REMARKS OF ANALYSTS.

F. P. Veitch.—The nitric-acid method has given in my hands results nearly as high as the peroxid method. The method was modified somewhat with a view to hastening the destruction of the organic matter. After adding potassium nitrate and burning some time the dish was cooled and a few cubic centimeters of nitric acid added, dried rapidly, and ignited cautiously. This gave a perfectly white ash and was much more rapid than the prescribed procedure. As much organic matter remains after the treatment with nitric acid, repeated treatment with nitric acid or aqua regia might give higher results.

In the peroxid method the silver crucible was strongly attacked in fusing sample No. 1. Some silver remained in solution even after evaporation and filtration. It was partly precipitated by dilution, but some remained in solution and was precipitated with the barium sulphate, which was purified by fusion with sodium carbonate, washed with water, dissolved in nitric acid, and reprecipitated with sulphuric acid. The precipitate in sample No. 2 was not contaminated by silver. In the present form I regard the peroxid method as the more accurate of the two methods. The correction for the sulphur in the peroxid was subtracted before reporting the results.

R. W. Thatcher.—I attempted to make the estimations of sulphur by the peroxid method, but only succeeded in driving everybody out of my end of the building, since our hoods are not yet completed sufficiently to carry off the fumes. The method seems to me to be a very undesirable one. In the first place, my sodium peroxid contains sulphur, and it is almost impossible to control the amount of sulphur by blank determinations since some sputtering of the material when water is first added is almost unavoidable. This fact along with the extreme difficulty of manipulation makes the method a very unsatisfactory one in my opinion.

W. G. Morrison.—The peroxid method was followed, nickel crucibles being used. The action was violent and there was an inclination toward spattering when sodium peroxid was added, and nickel appears every time in the filtrate on first filtering.

G. S. Fraps.—In the peroxid method a nickel or silver crucible of about 40 cc capacity gives more satisfactory results than a silver dish. Nickel is preferable, since silver was dissolved in every determination made by the writer. The silver chlorid settled out when the liquid was diluted and allowed to stand, and it then could be filtered off. If the organic material is added to the sodium hydroxid before the water has been completely driven off, the mixture is very likely to foam over, and considerable foaming takes place in any event. It is therefore advisable to continue heating the peroxid for a few minutes after the water has apparently been driven off, then stir the organic matter in quickly, and heat cautiously until danger of foaming has passed.

A blank determination is always necessary, even with chemically pure peroxid. At its best the fusion method is very slow. It is possible that an iron crucible could be used in place of a silver or nickel one.

CONCLUSIONS.

There is no doubt in the mind of the referee that the nitric-acid method gives results slightly lower than the peroxid method. It is possible that the former may be so modified as to give higher results. Owing to the difficulty of manipulating the peroxid method and the disinclination of some chemists to work with it, the referee believes that it should be subjected to further study before adoption as a provisional method in place of the nitric-acid method.

DETERMINATION OF SULPHATES.

The referee had some difficulty in obtaining a sample suitable for a study of the method of determining sulphates in plants. None of the materials available seemed to contain appreciable quantities of sulphates. The referee last year reported on the determination of sulphates in a few samples, all of which were found to contain an exceedingly small proportion of inorganic sulphur. Sorghum and teosinte were examined and gave no visible precipitate of barium sulphate. Cotton seed were caused to germinate, but the germinated seed contained only 0.064 per cent of sulphates, and this method of preparing the samples did not appear promising.

The sample for analysis was prepared as follows: One gram of potassium acid sulphate was dissolved in 100 cc water, mixed thoroughly with 200 grams of sorghum until the sorghum was all damp, sifted, dried, and again sifted several times. It is believed that a uniform sample was obtained by this method.

The method for sulphates has been described. It is the method described in the report of the referee last year. The results of the different analysts are presented in the following table:

TABLE III.—*Determination of sulphates.*

Analyst.	Per cent.
F. P. Veitch, Washington, D. C	0.400
Frank T. Shutt, Ottawa	.375
	.376
R. W. Thatcher, Washington (State)	.349
	.324
G. S. Fraps, Texas	.379
	.381
	.380
	.367

CONCLUSIONS.

The method seems to give fairly satisfactory results in the hands of the different analysts, and while the amount of sulphates in agricultural products seems to be, in general, very small, it may be well to have a method for their determination.

RECOMMENDATIONS.

The referee makes the following recommendations:

(1) That the method for the determination of sulphates, as already described, be printed as a provisional method.

(2) That the peroxid method of determining sulphur be subjected to further study, with a view to substituting it for the nitric acid method.

The **PRESIDENT.** Is there any discussion of the paper presented, or of the subject of ash?

Mr. **SHUTT.** As associate referee, I tried the sodium peroxid method, and wish to indorse what is said in regard to the unsuitability of that method for the determination of sulphur. It is disagreeable, sloppy, and incorrect, and it does not seem at all likely that we shall ever be able to make a good working method of it. I scarcely think it will be of any value to continue this investigation. The whole method is one that seems to me quite unsuitable for good and accurate work.

Mr. **PATRICK.** If it were true that it were impossible to obtain sodium peroxid free from sulphur, I should agree with the view that it is hardly worth while to continue these experiments; but we have at the Bureau of Chemistry quite a quantity of this reagent entirely free from sulphur, and we are doing very good work with the process. I admit all that has been said concerning the troublesomeness and disagreeableness of the method.

The **PRESIDENT.** In the absence of the referee on tannin the report on insecticides will be received.

REPORT ON INSECTICIDES AND FUNGICIDES.

By J. K. HAYWOOD, *Referee.*

The work for this year upon insecticides and fungicides consisted of the testing of various methods outlined in previous years, together with a discussion of new methods published since the last report of the referee.

Samples of Paris green, London purple, copper carbonate, potassium cyanid, soda lye, soap, tobacco extract, and formalin were forwarded early in the spring to those who expressed a desire to cooperate in the work. The methods of analysis used were, in the main, the same as those of last year, with the addition of such changes as previous work had shown to be advisable. A new method for determining formaldehyde was also added, and the gravimetric hexa-methyl-tetramine method, which had been shown to be practically worthless, was dropped.

Results on Paris green were received from eight chemists, including the referee; on London purple from eight chemists; on copper carbonate from four chemists; on potassium cyanid from four chemists; on soda lye from three chemists; on tobacco extract from two chemists; and on formalin from three chemists.

Paris Green.

Following are the results obtained upon Paris green and the comments of the various analysts:

Table I.—*Paris green.*

Analyst.	Moisture.	Total arsenious oxid.		Total copper oxid.			Soluble arsenious oxid.	
		Method I.	Method II.	Method I.	Method II.	Electrolytic.	Method I.	Method II.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
James Emery, Biochemic Div., U. S. Dept. Agr	1.00	60.00	53.30	27.74	30.67	....	7.04	....
	1.02	60.20	53.62	26.93	30.55	....	6.14	....
J. M. Price, Biochemic Div., U. S. Dept. Agr	....	....	53.77	27.53	30.64	....	7.06	....
	....	....	53.77	27.60	....	....	7.06	....
R. W. Thatcher, Pullman, Wash	.56	60.04	53.00	29.38	29.14	....	7.09	7.88
	.55	59.96	55.65	29.55	29.25	....	7.03	7.73
H. J. Warner, Bur. of Chem., U. S. Dept. Agr	....	60.15	55.44	28.31	28.61	....	7.52	9.85
	....	60.25	55.69	28.14	28.77	....	7.42	9.85
R. J. Davidson and J. B. McBryde, Blacksburg, Va	.54	....	53.49	28.84	29.18	28.78	6.83	6.03
	.53	....	53.64	28.74	29.10	28.97	6.82	5.97
	....	....	53.66	28.88	29.26	28.90	....	....
	....	....	53.53	28.87	....	....	....	....
A. G. Ford, Stillwater, Okla	.85	59.61	53.39	28.03	27.81	....	7.21	....
	1.00	59.86	53.49	28.03	28.20	....	7.50	....
	....	59.61	53.25	28.28	28.29	....	7.21	....
	....	59.67	53.25	28.03	....	....	....	....
W. P. Allen, New Brunswick, N. J.	1.31	59.52	....	27.72	27.93	....	7.16	8.29
	1.25	59.58	....	27.82	27.98	....	7.17	8.29
	....	....	....	27.84	28.08	....	7.27	7.67
	....	....	....	....	28.09	....	....	....
	....	....	....	....	28.21	....	....	....
	....	....	....	....	28.26	....	....	....
J. K. Haywood, Bur. of Chem., U. S. Dept. Agr	.70	59.94	54.92	28.24	28.95	28.63	7.20	8.07
	.63	59.87	54.13	28.33	29.04	....	7.12	8.31

COMMENTS BY ANALYSTS.

R. W. Thatcher.—In Method II for total arsenious oxid I stirred for some time in an attempt to dissolve all the free arsenious oxid but did not get all the crystals to dissolve, however. I then used a modification suggested by Avery, as follows: A weighed sample of the green was boiled with sodium acetate as in the determination of soluble arsenic by his method, the mixture filtered through asbestos, the soluble arsenic determined in the filtrate and the residue on the asbestos dissolved in dilute hydrochloric acid and titrated according to the Avery-Beans method. The total arsenic figures determined in this way were 59.90 and 59.84 per cent. I found in Method I for soluble arsenic that as the weight of green taken varied, the soluble arsenic varied according to the following figures: One gram of green gives 7.09 and 7.03 per cent soluble arsenious oxid; 0.809 gram green gives 7.29 per cent soluble arsenious oxid; 0.484 gram green gives 7.51 per cent soluble arsenious oxid.

R. J. Davidson and *J. B. McBryde.*—In method II for total arsenious oxid varying results were obtained, according to the amount of hydrochloric acid added, and the time of stirring. The results sent in by us were obtained by using 1 cc of hydrochloric acid to each determination and stirring frequently for 10 minutes; an N/20 iodin solution was used.

In method I for total copper oxid the results were obtained by using 3 grams of potassium iodid to each determination. With only ten times the weight of copper, it was found impossible to obtain concordant results. An N/20 thio solution was used throughout.

In method II for total copper oxid N/10 potassium cyanid was used.

A. G. Ford.—In method II for total arsenious oxid a white residue remained, which did not go into solution, even on the addition of 15 drops of hydrochloric acid and 24 hours' standing. I therefore filtered off the solution and washed the residue with cold water; placed the filter paper in a 250 cc flask (along with blank determination), digested 10 minutes with 25 cc of 2 per cent sodium hydrate, added 25 cc of hydrochloric acid and 3 grams of potassium iodid after cooling to 80°, and allowed to stand 15 minutes; diluted, decolorized with thio sulphate, made alkaline with soda, then acid with hydrochloric acid, and finally added excess of sodium bicarbonate and titrated with standard iodin. By adding the figure thus obtained to the previous figures the result was 59.93 per cent arsenious oxid.

W. P. Allen.—In method II for total copper oxid the potassium cyanid solution must be added very slowly or more will be used than is necessary.

J. K. Haywood.—Method II for total arsenious oxid, as would be expected from last year's work, gave low results. I would suggest either of the following modifications of this method; (*a*) Pulverize the sample and weigh out about 0.3 to 0.4 grams in a beaker, add 25 cc of water and, while constantly stirring, add concentrated hydrochloric acid a drop at a time until all Paris green is in solution and free arsenious oxid remains as a residue. Filter and wash the residue and determine the arsenious oxid in the filtrate according to the regular Avery-Beans method. The filter and contents are washed back into the beaker, 5 grams of sodium bicarbonate added, and the solution boiled until all arsenious oxid has dissolved. The resulting solution is cooled, slightly acidified with hydrochloric acid, and made alkaline again with sodium bicarbonate and finally titrated with iodin. (*b*) Take 0.4 gram of the finely ground green and boil with 25 cc of sodium acetate (containing 12 grams) for 10 minutes. Then add concentrated hydrochloric acid a drop at a time until solution is effected (about 10 cc of the acid will be necessary). Add concentrated sodium carbonate solution a drop at a time until a slight precipitate appears, then add a solution containing 2 to 3 grams of sodium potassium tartrate and finally sodium bicarbonate in excess. Titrate with iodin in the usual manner. By method (*a*) 59.87 per cent of total arsenious oxid was obtained and by method (*b*) 59.62 per cent.

Method I for total copper oxid appears to give results somewhat lower than the truth and method II somewhat higher results.

DISCUSSION OF THE RESULTS.

It will be at once seen that method I for total arsenious oxid has given most excellent results, as it did last year. Method II has given low and variable results, as was also to be expected from previous work. There is no doubt that method II would prove of great value if modified to include free arsenious oxid. Your referee has therefore spent some time working upon this method and suggests the above modifications, which give most excellent results. Others have evidently seen the advisability of changing this method, as in the comments of the analysts given above two other modifications are suggested, one by R. W. Thatcher which was originally suggested to him by Mr. Avery, and one by A. G. Ford. Both of these modifications evidently give good results.

Just after the method above, marked (*a*), was sent to press your referee entered into correspondence with Mr. Avery, one of the originators of the method, and found that he was working along the same line and had also sent a modification of his method to the publishers. Mr. Avery was kind enough to forward two modifications that he had worked out. They are as follows:

(*c*) Weigh out a small quantity of the finely ground green and treat with N/2 hydrochloric acid, from 5 to 10 cc for each 0.1 gram of the green present. Boil gently; if solution is not effected add a cold saturated solution of sodium acetate, using about 3 grams for each 0.1 gram of the green, and boil until all arsenious oxid is dissolved. Now add alkaline tartrate and solid bicarbonate and after dilution titrate in the ordinary way.

(*d*) Use the method as given above by Mr. Thatcher, which was originally suggested by Mr. Avery.

Your referee tested both of these methods and found that they gave most excellent results—60.30 per cent by the former and 60.04 per cent by the latter. After receiving the two methods from Mr. Avery the referee then thought of the other modifications marked (*b*) above. Since the modifications proposed by Mr. Avery were of great value to him in working out method (*b*), he feels that this method is really a joint production with Mr. Avery rather than any original idea of his own.

It will thus be seen that four modifications are given above, each of which gives excellent results. The one to be used would best be determined by the exigencies of the case.

While the results obtained with method I for total copper oxid by any one analyst agree with each other very well, the averages of the results obtained by different chemists vary quite a good deal, and this seems to be true also for method II. Judging from all the results there appears to be a marked tendency in the direction of low results for method I and high results by method II. There are a few exceptions to this—one in the case of Mr. Thatcher, who obtains higher results by method I than by method II, and one in the case of Mr. Ford, who obtained practically the same figures by each method. Since it is only by averages, however, that one can judge, the referee is inclined to believe that it would be best to adopt the electrolytic as the official method and allow the others for the present to remain as provisional, to be used by those who obtain as good results with them as with the electrolytic method.

A comparison of the figures obtained by the individual analysts and the averages given by the various chemists using method I for soluble arsenious oxid shows a fair agreement when the difficulty of separating the free from the combined arsenious oxid is considered. The results obtained by method II for soluble arsenious oxid are in the main higher than those obtained by method I. This is what would be expected from previous work. The agreement between these results leaves a good deal to be desired and plainly shows that this method, which at the best is only approximate, must be carried out under rigid rules in order to get reliable results.

LONDON PURPLE.

TABLE II.—*London purple.*

Analyst.	Moisture.	Total arsenious oxid.	Total arsenic oxid.	Soluble arsenious oxid.	Soluble arsenic oxid.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
James Emery, Biochemic Div., U. S. Dept, Agr	....	12.61	30.59	....	....
	....	12.68	....	....	....
J. M. Price, Biochemic Div., U. S. Dept. Agr	....	12.98	29.79	....	....
R. W. Thatcher, Pullman, Wash	0.95	12.65	29.78	3.61	9.37
	.93	12.63	30.00	3.61	9.30
H. J. Warner, Bur. of Chem., U. S. Dept. Agr	....	12.92	30.19	2.58	6.67
	....	....	....	2.58	6.53
	....	....	....	2.81	6.67
R. J. Davidson and J. B. McBryde, Blacksburg, Va.	2.12	12.77	29.68	2.48	6.66
	2.19	12.67	29.74	2.48	6.61
	....	....	....	2.48	6.72
	....	....	....	2.48	6.69
A. G. Ford, Stillwater, Okla	1.71	12.72	32.26	3.04	4.62
	1.71	12.72	....	3.04	....
W. P. Allen, New Brunswick, N. J	2.39	11.62	31.56	1.62	....
	2.41	11.71	31.56	1.85	....
	....	11.75	31.56	1.97	....
J. K. Haywood, Bur. of Chem., U. S. Dept. Agr	2.44	13.03	29.70	2.37	6.27
	2.26	13.03	29.70	2.61	6.67
	....	....	....	2.61	6.34

COMMENTS BY ANALYSTS.

R. J. Davidson and J. B. McBryde.—In making all iodin titrations N/20 iodin was used. In determining the total arsenic oxid it is very difficult to read the end point with thio-sulphate. We therefore suggest that some method of getting rid of part of the coloring matter could be used to advantage. One way would be to use 200 cc of the solution and proceed as under total arsenious oxid until the solution is filtered off, then acidify a portion with hydrochloric acid at 80° and proceed as under arsenic oxid. Another way would be to use 100 cc instead of 50 cc, as under total arsenic oxid, make alkaline with sodium carbonate and make up to a definite volume. Filter off half of the solution, acidify with hydrochloric acid, heat to 80°, and proceed as under total arsenic oxid.

A. G. Ford.—In determining total and soluble arsenic oxid I found it extremely difficult to read the end reactions with thio-sulphate and iodin within a range of 0.3 to 0.5 cc.

DISCUSSION OF RESULTS.

A glance at column 2 shows that the results obtained for total arsenious oxid in London purple agree with each other extremely well, with possibly one exception.

The results obtained by six different chemists upon total arsenic oxid agree very well with each other, while two chemists obtained high results.

Your referee is loath to drop this method of examining London purple since it is the only method known to him for determining both forms of arsenic in this material, and, moreover, the figures for this and previous years show that a majority of the chemists using it get very reliable results. The referee also knows from a personal experience of two or three years that when one is familiar with the method very accurate results can be obtained. It is of course difficult to read the end points without previous experience, therefore any modification of this method which would so far rid the solution of coloring matter as to make the readings easy, even for the inexperienced, would be of great value. Two such modifications have been suggested by Messrs. Davidson and McBryde in their comments given above. The referee is therefore of the opinion that no better work could be performed during the coming year by the referee on insecticides and fungicides than a testing of the modifications given by Messrs. Davidson and McBryde and such other modifications as might suggest themselves.

The results obtained for soluble arsenious oxid by four chemists agree fairly well with each other. One chemist obtains slightly low results and one high results. The figures for soluble arsenic oxid show an excellent agreement in three cases, while in the other two cases they vary considerably. These two methods of examination also can only lead to reliable results by following very strict rules of procedure, just as in the determination of crude fiber. It has been the experience of the referee that results within about 0.25 to 0.75 per cent of each other (according to the amount of soluble arsenious and arsenic oxid present) can be obtained by these two methods, which are exact enough for all practical purposes, although more correct figures are greatly to be desired for scientific purposes. It might be well for someone to undertake work to determine whether a shorter time of standing would be long enough for all of the soluble arsenious and arsenic oxids to go into solution and whether more concordant results would not be obtained by a shorter period of standing.

COPPER CARBONATE.

TABLE III.—*Copper carbonate.*

Analyst.	Total copper oxid.		
	Method I.	Method II.	Electrolytic method.
	Per cent.	*Per cent.*	*Per cent.*
H. J. Warner, Bur. of Chem., U. S. Dept. Agr.	59.50	59.25	....
	59.50	59.25	....
R. J. Davidson and J. B. McBryde, Blacksburg, Va.	59.12	58.42	58.31
	58.88	58.22	....
A. G. Ford, Stillwater, Okla.	58.57	58.71	....
	58.77	59.10	....
	58.06	58.71	....
	58.57	58.32	....
J. K. Haywood, Bur. of Chem., U. S. Dept. Agr.	58.11	59.67	58.73
	58.48	59.67	....

COMMENTS BY ANALYSTS.

R. J. Davidson and J. B. McBryde.—In method I N/20 thio-sulphate was used, but even with this strength a difference of reading of 0.05 cc gave a difference of more than 0.2 per cent in the results. In method II N/10 potassium-cyanid solution was used, but, as in the case of thio, a difference of 0.1 cc gave a difference of 0.2 per cent in the results.

J. K. Haywood.—As in the determination of total copper in paris green I obtained slightly low results by the thio-sulphate method and high results by the potassium-cyanid method.

DISCUSSION OF THE RESULTS.

A discussion of these figures is rather difficult, as no general law, such as was shown by the figures for total copper in Paris green, is brought out. One chemist appears to get slightly high results by both methods, one to get high results by method I and correct results by method II, one to get equally correct results by both methods and one to get slightly low results by method I and extremely high results by method II. It would therefore appear that, as in the case of Paris green, it would be best to adopt the electrolytic as the official method and leave the other two methods as provisional, to be followed or not, according as the individual chemist deems it advisable upon comparing the results so obtained by him with those given by the standard electrolytic method. From the remarks of Messrs. Davidson and McBryde, it appears extremely likely that a greater part of the variation in the above figures was caused by too great a strength of the standard solution. A valuable piece of work would be performed if the referee for the coming year could work out just what the factors are causing the slight variation in the results obtained by different chemists.

POTASSIUM CYANID.

TABLE IV.—*Potassium cyanid.*

Analyst.	Cyanogen.		Chlorin.
	Volumetric.	Gravimetric.	
	Per cent.	*Per cent.*	*Per cent.*
James Emery, Biochemic Div., U. S. Dept. Agr	39.42		8.83
	39.32		
T. M. Price, Biochemic Div., U. S. Dept. Agr	39.32		
H. J. Warner, Bur. of Chem., U. S. Dept. Agr	39.46		
	39.60		
R. J. Davidson and J. B. McBryde, Blacksburg, Va	39.90		
	39.78		
J. K. Haywood, Bur. of Chem., U. S. Dept. Agr	40.49	40.42	8.98
	40.49		
	40.11		

COMMENT BY ANALYSTS.

R. J. Davidson and J. B. McBryde.—Two separate portions were weighed out for analysis, and the titrations of the two samples agree within 0.05 cc. The difference in percentage is 0.3 per cent. In other words, one drop of N/10 silver solution is equal to 0.3 per cent potassium cyanid. Duplicate titrations with N/10 iodin gave 39.80 per cent of cyanogen.

J. K. Haywood.—I first determined the cyanogen according to directions. This method is based on the following equation:

$$2KCN+AgNO_3=KCNAgCN+KNO_3.$$

In other words, we might say that only one-half of the potassium cyanid is titrated. I then added potassium chromate and continued the titration until the appearance of the usual brown color of silver chromate. This last titration not only gives the other half of the cyanogen according to the equation $KCNAgCN+AgNO_3=KNO_3+2\,AgCN$, but gives any chlorin that may be present as silver chlorid. Therefore, if we represent the number of cubic centimeters used in the first titration by M and the number used in the second titration by N, then N−M=the number of cubic centimeters used by chlorin, and 2 M is the number used by the cyanogen according to the following equation:

$$2KCN+2AgNO_3=2KNO_3+2AgCN.$$

Having thus determined the chlorin, the total amount of silver chlorid and silver cyanid was determined gravimetrically and from this was subtracted the amount of silver chlorid corresponding to the chlorin figure, thus leaving the silver cyanid, from which the result 40.42 per cent of cyanogen was obtained.

DISCUSSION OF THE RESULTS.

The referee sent out this sample thinking that it was a pure sample of potassium cyanid, but found out soon after that it was a mixture of sodium and potassium cyanids, sodium chlorid, and other ingredients in such proportions that just about the amount of cyanogen was present as would be present in chemically pure potassium cyanid. This explains the chlorin figures given by both the referee and the associate referee. Of course the presence of this chlorin would not interfere in any way with the volumetric determination of the cyanogen present. The results obtained show excellent agreement, with the possible exception of the referee's figures, which are rather high. This is undoubtedly due to the fact that the referee worked upon the sample when it was first put up, while the other chemists worked upon it some months later, when the cyanid, which is very hygroscopic, had gained in moisture. On account of the excellent agreement of the results obtained during the last two years' work, and also because the gravimetric determination shows that the volumetric is correct, it would seem that this method ought to be adopted as one of the official methods, with one change, however, and that is, an N/20 instead of an N/10 silver nitrate solution should be used.

SODA LYE.

.ABLE V.—*Soda lye.*

Analyst.	Sodium hydroxid.		Sodium carbonate.	
	Method I.	Method II.	Method I.	Method II.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
H. J. Warner, Bur. of Chem., U. S. Dept. Agr	73.44	73.16	4.47	4.60
	73.44	73.16	4.47	4.60
R. J. Davidson and J. B. McBryde, Blacksburg, Va	73.98	71.57	3.91	5.53
	73.58	71.57	4.15	4.96
J. K. Haywood, Bur. of Chem., U. S. Dept. Agr	73.59	72.87	4.20	4.13
	73.59	72.87	4.20	4.13

COMMENTS BY ANALYSTS.

R. J. Davidson and J. B. McBryde.—Under method I for sodium hydroxid, tests were made with the portion to which barium chlorid was added, in order to determine the possibility of titrating the solution without removing the precipitate of barium carbonate. As soon as barium chlorid was added to the solution samples were drawn off and titrated. The results obtained in this way were identical with those obtained from decanted and filtered samples. Our tests go to show that the removal of the barium carbonate from the solution is unnecessary.

We also made tests to determine the possibility of using only one indicator under method I. After the addition of barium chlorid, methyl orange gave results identical with those obtained when using phenolphthalein. By using phenolphthalein in boiling solution we obtained the same results in total alkalies as when using methyl orange.

DISCUSSION OF RESULTS.

A consideration of this table shows that all of the chemists obtained closely agreeing results on sodium hydroxid, and very fair results on sodium carbonate, by method I. The results on these two constituents by method II do not agree nearly so well. Just as in last year's work, it will be noted that all of the analysts obtained higher figures for sodium hydrate by method I than by method II, and lower results for sodium carbonate by method I than by method II. Your referee gave last year his reasons for believing that the results obtained by method II were more accurate than those obtained by method I, but it does not appear from either year's work that method II is as easy of execution as method I. It is suggested that the only way of telling which is the better method is by making an actual gravimetric determination of the carbon dioxid present. This should be done during the coming year, so that method I, which now appears to give the most concordant results, may either be adopted as an official method or rejected. In case method I is found to give low results on sodium carbonate, method II could be further worked upon to find the cause of the variations in results by different chemists. It would appear from the work of Messrs. Davidson and McBryde that a removal of the precipitated barium carbonate in method I is not necessary. This, if true, is a very valuable point, in that it greatly shortens the time necessary for making a determination.

TOBACCO EXTRACT.

TABLE VI.—*Tobacco extract.*

Analyst.	Nicotin.	
	Method I.	Method II.
	Per cent.	*Per cent.*
R. J. Davidson and J. B. McBryde, Blacksburg, Va	22.37	22.92
	22.64	22.69
	23.08	21.94
	....	21.39
J. K. Haywood, Bur. of Chem., U. S. Dept. Agr	23.53	21.93
	23.70	21.88
	22.70	21.74

COMMENTS BY ANALYSTS.

R. G. Davidson and J. B. McBryde.—In method II the substance was weighed out in small beakers and allowed to stand, one 24 hours, one 36 hours, and two for 48 hours.

J. K. Haywood.—In both methods I and II it was found necessary to distill over about 500 cc of water in order to obtain all the nicotin. In method II an attempt was made to drive off all ammonia by heating to 50°–60° C. after adding the sodium hydroxid, but a great loss of nicotin also took place. It was also found that the longer the extract was allowed to stand in the open in contact with sodium hydrate, the smaller the figure obtained for nicotin, showing that there was a constant loss of this substance.

DISCUSSION OF THE RESULTS.

From the work done last year it was evident that the Lloyd method gave low results upon extracts containing a large amount of nicotin. It was also found that the Winton method (II) gave extremely high results in case large amounts of ammonia and ammonium salts were present, unless the sodium hydroxid was allowed to stand in contact with the extract for a very long period. This year's results show that if large quantities of ammonium salts and ammonia are not present fair results can be obtained by method II, but there is a continuous loss of nicotin the longer the extract stands in the open in contact with the sodium hydrate. It is manifestly impossible to tell with all the extracts the exact point at which all the ammonia is given off and no nicotin has yet volatilized. In fact, such a point very likely does not exist. It would therefore appear that both the Lloyd method and method II must be rejected, which leaves only method I. Last year very fair results were obtained by this method, considering the difficulty of the determination, and the experience has been repeated this year. Since this is so, and the Kissling method has stood the test of time when used by various chemists outside of the association, your referee is of the opinion that it should be adopted as the official method of the association, with an understanding, however, of its limitations, the principal of which is that a large part of the pyridin present is determined as nicotin.

FORMALIN.

TABLE VII.—*Formalin.*

Analyst.	Formaldehyde.		
	Method I.	Method II.	Method III.
	Per cent.	*Per cent.*	*Per cent.*
J. K. Haywood, Bur. of Chem., U. S. Dept. Agr.	18.82	18.96	18.58
	18.96	18.81	18.68
B. H. Smith, Bur. of Chem., U. S. Dept. Agr	18.86	18.80	18.40
	18.93	18.86	18.16
	18.92	....	....
F. F. Ladd and C. H. Kimberly, Agricultural College, N. Dak.	22.4	21.0	18.99
	19.4	21.0	18.94
	22.0	18.0	19.08

COMMENTS BY ANALYSTS.

Messrs. Ladd and Kimberly.—Method I gives varying results according to the length of time the sample stands. We believe the results to be high. Method II seems open to serious objections, since a slight error in reading causes a large error. A difference of 0.1 cc may make an error of 3 per cent in strength. Method III has always given the most trustworthy results for laboratory use, and rosolic acid is a more satisfactory indicator than litmus. No corrections have been made for free acids present.

DISCUSSION OF THE RESULTS.

From the table above it appears that two chemists were able to get most excellent comparative results by both methods I and II, while a third chemist obtained widely varying results by these two methods. For method III the first two chemists obtained low results, while the third chemist obtained very accurate results. The referee can not agree with Messrs. Ladd and Kimberly in their conclusion that method I gives unreliable, and method III extremely reliable, results. It has been his experience that method I gives very excellent results, while method III always gives low results, and is further open to the objection that it is a very hard matter to read the end point. It is perfectly true that method II is open to the objection that a very slight error in the reading causes a large error in the percentage, and it would appear that method II should only be used upon very dilute solutions of formaldehyde.

It might be well to say just here that method II is not the same as method II of last year, which was replaced by the present method proposed by Romijn,[a] and is as follows:

Fifteen cubic centimeters of N/10 silver nitrate are treated with six drops of 50 per cent nitric acid in a 50 cc flask; 10 cc of a solution of potassium cyanid (containing 3.1 grams of potassium cyanid in 500 cc of water) are then added and well shaken.

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[a] Zeit. anal. Chem., 1897, **86**: 18–24.

An aliquot portion of this filtrate, say 25 cc, is titrated according to the method of Volhard with an N/10 solution of ammonium-sulpho-cyanate for excess of silver. Another 15 cc portion of N/10 silver nitrate is acidified with 50 per cent nitric acid and treated with 10 cc of the potassium cyanid solution to which has been added a weighed quantity of the dilute formaldehyde solution. The whole is made up to 50 cc and a 25 cc filtrate from it is titrated with N/10 ammonium-sulpho-cyanate for the excess of silver as before. The difference between these results multiplied by 2 gives the amount of potassium cyanid that has been used by the formaldehyde in terms of N/10 ammonium-sulpho-cyanate. Each cubic centimeter of this is equal to 3 mg of formaldehyde.

Since this difference of opinion exists between your referee and two of the chemists who were engaged in the work, it would appear advisable to examine the opinions that have been published on the subject.

C. Wallnitz, in an article[a] which appeared early in 1903, compared several methods for determining formaldehyde. He found that the hydrogen peroxid (method I) and the iodin methods were superior to the others tested when the reagents are freshly prepared, and that the Legler method (method III) lacked distinctness in the end point reading.

B. H. Smith, in an article[b] published during the fall of 1903, found that the hydrogen peroxid method (method I) is very satisfactory for strong solutions; that the Legler method (method III), while not giving quite as high results, is fairly satisfactory, and that the potassium cyanid method (method II) gives good results on dilute solutions.

The referee will, therefore, make no recommendations concerning the adoption of any of these methods, but will leave them until the work of the association definitely establishes the superiority of one or the other.

RECOMMENDATIONS.

I would respectfully recommend:

(1) That method I for total arsenious oxid in Paris green be adopted as an official method, this being a repetition of my last year's recommendation.

(2) That the four modifications of the Avery-Bean method for total arsenic in Paris green be tested to find which is the best modification to be adopted as an optional official method.

(3) That the electrolytic method be adopted as the official method for determining copper in Paris green and in copper carbonate, and that the volumetric methods be further tested, paying particular attention to the remarks of Messrs. Davidson, McBryde, and Allen upon this subject.

(4) That an attempt be made to so change the procedure in the analysis of London purple that some of the organic matter may be removed. The remarks of Messrs. Davidson and McBryde are of value in this connection.

(5) That the volumetric silver nitrate method be adopted as the official method of determining cyanogen in potassium cyanid and that an N/20 instead of an N/10 solution of silver nitrate be used.

(6) That the methods of examination of soda lye be further tested and compared with the gravimetric methods and that points brought out by Messrs. Davidson and McBryde be given consideration.

(7) That the Kissling method for determining nicotin be adopted as an official method.

(8) That especial attention be given to the methods for determining formaldehyde in order to avoid needless repetition of work.

I desire to express my thanks to those gentlemen who have so kindly and ably cooperated with me in the above work, and especially to Messrs. Davidson and McBryde for many valuable suggestions.

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[a] Zeit. angew. Chem., April 7, 1903.

[b] J. Am. Chem. Soc., 1903, vol. 25, No. 10.

(A discussion by Messrs. Davidson, Mewborne, Hird, and Haywood on the subject of nicotin determinations, especially in strong extracts, followed the reading of the report. The points discussed are brought out in the report as printed in full.)

The **President**. In the absence of the referee on tannin the report of the committee on fertilizer legislation will be received.

REPORT OF COMMITTEE ON FERTILIZER LEGISLATION.

By H. W. Wiley, *Chairman.*

As it was not practicable to call a meeting of the committee on fertilizer legislation, I corresponded with each member of the committee to secure an expression of opinion, first, as to the necessity of legislation regulating interstate commerce and foreign commerce in fertilizers and fertilizer ingredients; and, second, in regard to the character that such legislation should assume. I have received replies from members of the committee, and present the correspondence for publication.

Washington, D. C., *October 29, 1903.*

Dear Sir: The necessity for some national legislation in regard to fertilizers is becoming more and more evident every day. The States, of course, have legislation controlling commerce wholly within the States, but their authority does not extend beyond the State line. The multiplicity of labels and regulations in the different States is a real annoyance to manufacturers and to the trade.

It is believed that national legislation controlling interstate commerce in fertilizers and commerce therein in the District of Columbia and the Territories and regulating foreign commerce therein would prove beneficial in many ways. Such legislation, if wisely drawn, would serve as a model in the different States and would gradually secure a greater uniformity in State laws, thus removing unnecessary restrictions on trade, both in manufacture and in transit.

The committee on fertilizer legislation of the Association of Official Agricultural Chemists should make some recommendations at the coming meeting respecting national legislation. The committee doubtless could also properly make suggestions in regard to uniform State legislation. I therefore ask that you express your opinion on the following points:

(1) Is national legislation of the kind indicated above desirable?

(2) If so, suggest tentatively the form of an act which should be brought before the Congress of the United States.

(3) State how, in your opinion, national legislation should influence State legislation in regard to uniformity and freedom from unnecessary restrictions on commerce.

(4) Miscellaneous matters respecting fertilizer legislation not included in the above items.

I shall be glad to have a full expression of your views on these points and earnestly request that you place this expression in my hands not later than November 14, proximo, in order that the chairman may have an opportunity to collate and present the views of the different members of the committee to the association.

Respectfully,

H. W. Wiley,
Chairman Committee on Fertilizer Legislation, A. O. A. C.

Raleigh, N. C., *October 30, 1903.*

Dear Sir: I have your favor of the 29th in regard to national fertilizer legislation. I think some legislation along this line is probably desirable. The only difficulty I see is that in case the bill went into details it might give trouble in some of the States. You will remember that we had considerable difficulty in agreeing on the names by which to designate the fertilizing constituents in our committee a few years ago. Some ten of the Southern States have practically agreed on a uniform law, which was framed by the State chemists and commissioners of agriculture in an association known as the Cotton States Association of Commissioners of Agriculture. A copy of this law is to be found on page 52 of the proceedings of this association, which I am sending under separate cover.[a] In this association we had difficulty in agreeing on names by which to designate the constituents. The main features of this bill, that is, the portions relating to uniform branding and guaranties, is now in operation, or will be in operation in Virginia, North Carolina, Tennessee, Alabama, Georgia, Louisiana, and Arkansas, with prospects that Florida and South Carolina will conform to it. I think the details of inspection and the control of the fertilizer trade should be left to each individual State. While our bill includes all the minutiæ, it was understood in our discussion of it that each State was at liberty to arrange the minor details to suit conditions.

B. W. KILGORE, *State Chemist.*

FERTILIZER LAW DRAFTED BY COMMITTEE OF THE COTTON STATES ASSOCIATION OF COMMISSIONERS OF AGRICULTURE.

A BILL to be entitled an act to regulate and make uniform with the acts of other States of the Union the registration, sale, inspection, and analysis of commercial fertilizers, acid phosphates, fertilizer materials, and chemicals, in the State of ——, and to consolidate all laws relating to said sales, inspection, and analysis, and to repeal all other laws, or parts of laws, in conflict therewith.

SECTION 1. *Be it enacted by the general assembly of the State of* ——, That all manufacturers, jobbers, and manipulators of commercial fertilizers and fertilizer materials to be used in the manufacture of the same, who may desire to sell or offer for sale in the State —— such fertilizers and fertilizer materials, shall first file with ——, of the State of ——, upon forms furnished by said ——, the name of each brand of fertilizer, acid phosphate, fertilizer material, or chemical which they may desire to sell in the said State, either by themselves or their agents, together with the name and address of the manufacturer or manipulator; and also the guaranteed analysis thereof, stating the sources from which the phosphoric acid, nitrogen, and potash are derived, and if the same fertilizer is sold under a different name or names, said fact shall be so stated, and the different brands which are identical shall be named.

SEC. 2. All persons, companies, manufacturers, dealers, or agents, before selling or offering for sale in this State any commercial fertilizer or fertilizer material, shall brand or attach to each bag, barrel, or package the brand name of the fertilizer, the weight of the package, the name and address of the manufacturer, and the guaranteed analysis of the fertilizer, giving the valuable constituents of the fertilizer in minimum percentages only. These items shall be branded or printed on the package in the following order:

1. Weight of each package in pounds.
2. Brand name or trade-mark.
3. Guaranteed analysis.
4. Available phosphoric acid..per cent.
5. Nitrogen..per cent.
6. Potash..per cent.
7. Name and address of the manufacturer.

In bone meal, tankage, or other products, where the phosphoric acid is not available to laboratory methods, but becomes available on the decomposition of the products in the soil, the phosphoric acid shall be claimed as total phosphoric acid, unless it be desired to claim available phosphoric acid also, in which latter case the guaranty must take the form above set forth. In the case of bone meal and tankage, manufacturers may brand on the bags information showing the fineness of the product, provided it takes a form approved by the ——.

SEC. 3. If any commercial fertilizer or fertilizer material offered for sale in this State shall, upon official analysis, prove deficient in any of its ingredients, as guaranteed and branded upon the sacks or packages, and if by reason of such deficiency the commercial value thereof shall fall 3 per cent below the guaranteed total commercial value of such fertilizer or fertilizer material, then any note or obligation given in payment thereof shall be collectible by law only for the amount of the actual total commercial value as ascertained by said official analysis.

SEC. 4. Be it further enacted, that the words "High grade" shall not appear upon any bag or other package of any complete fertilizer which complete fertilizer contains, by its guaranteed analysis, less than 10 per cent available phosphoric acid, 1.65 per cent nitrogen (equivalent to 2 per cent of ammonia), and 2 per cent of potash, or a grade or analysis of equal total commercial value; that the word "Standard" shall not appear upon any bag or other package of any complete fertilizer which contains, by its guaranteed analysis, less than 8 per cent available phosphoric acid, 1.65 per cent nitrogen (equivalent to 2 per cent ammonia), and 2 per cent potash, or a grade or analysis of equal total commercial value; that the words "High grade" shall not appear upon any bag or other package of any acid phosphate with potash which shall contain by its guaranteed analysis less than 13 per cent available phosphoric acid and 1 per cent of potash, or a grade or analysis of equal total commercial value; that the word "Standard" shall not appear upon any bag or other package of any acid phosphate with potash which shall contain by its guaranteed analysis less than 11 per cent available phosphoric acid and 1 per cent potash, or a grade or analysis of equal total commercial value; that the words "High grade" shall not appear upon any bag or other package of any plain acid phosphate which shall contain by its guaranteed analysis less than 14 per cent available phosphoric acid; and, lastly, that the word "Standard" shall not appear upon any bag or other package of any plain acid phosphate which shall contain, by its guaranteed analysis, less than 12 per cent available phosphoric acid.

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[a] A copy of this law follows.

It is further hereby provided that no complete fertilizer, acid phosphate with potash, acid phosphate with nitrogen, or plain acid phosphate shall be offered for sale in this State which contains less than 12 per cent of total plant food, namely, available phosphoric acid, nitrogen, and potash, either singly or in combination, provided that in mixed fertilizers there shall not be claimed less than 1 per cent of potash and 0.82 per cent of nitrogen when one or both are present in the same mixture.

Sec. 5. Be it further enacted, that all manufacturers and manipulators, or agents representing them, who have registered their brands in compliance with section 1 of of this act, shall forward to ——— a request for tax tags, stating that said tags are to be used upon brands of fertilizers and fertilizer materials registered in accordance with this act, and said request shall be accompanied with the sum of ——— cents per ton as an inspection fee, whereupon it shall be the duty of ——— to issue tags to parties applying, who shall attach a tag to each bag, barrel, or package thereof, which, when attached to said package, shall be prima facie evidence that the seller has complied with the requirements of this act. Any tags left in the possession of the manufacturers shall not be used for another season, but shall be redeemed with new tags within sixty days after the close of the department's fiscal year, which fiscal year shall be comprised between the dates ———, inclusive. The color of the said tax tags must be changed each fiscal year, and there shall not be printed upon said tax tags any brand name or analysis.

Sec. 6. Be it further enacted, That it shall not be lawful for any manufacturer or company, either by themselves or their agents, to sell or offer for sale in this State any fertilizer or fertilizer material that has not been registered with ———, as required by this act. The fact that the purchaser waives the inspection and analysis thereof shall be no protection to said party selling or offering the same for sale.

Sec. 7. The guaranteed analysis of each and every brand of fertilizer or fertilizer material must, without exception, remain uniform throughout the fiscal year for which it is registered, and in no case, even at subsequent registration, shall the grade be lowered, although the proportion of the available constituents may be changed so that the decrease of one constituent may be compensated for in value by the increase of the other or others. Such proposed change must first receive the approval of the ———. A brand name (and) or trade-mark registered by one manufacturer shall not be entitled to registry by another, and the manufacturer having first registered and used the said brand name (and) or trade-mark shall be entitled to it, even should said brand name (and) or trade-mark not be offered for current registration at the time. Nothing in this section shall be construed as debarring the right of any manufacturer to establish his ownership in and prior right of registration of, any brand name (and) or trade-mark, whether said brand name (and) or trade-mark had been previously registered or not.

Sec. 8. No person, company, dealer, or agents shall sell, expose, or offer for sale in this State any pulverized leather, raw, steamed, roasted, or in any other form, either as a fertilizer or fertilizer material, without first making full and explicit statement of the fact in registration with the ——— and furnishing satisfactory proof that the nitrogen is sufficiently available and valuable for the purpose for which sold.

Sec. 9. Be it further enacted, That the ——— shall appoint as many inspectors of fertilizers as in said ——— judgment may be necessary, who shall hold their offices for such time as said ——— in ——— judgment shall think best for carrying out the provisions of this act. The greatest compensation that any one inspector of fertilizers shall receive shall be at the rate of ——— dollars per month and his actual expenses while in the discharge of his duty as such inspector. It shall be their duty to inspect all fertilizers, acid phosphates, chemicals, cotton-seed meal, or other fertilizing material that may be found at any point within the limits of the State, and go to any point when so directed by ——, and shall see that all fertilizers and fertilizer materials are properly tagged.

SEC. 10. Be it further enacted, That each of the inspectors of fertilizers shall be provided with bottles of not less than eight (8) ounces capacity in which to place samples of fertilizers and fertilizer materials drawn by him; and it shall be the duty of each inspector of fertilizers to draw, with such an instrument as shall secure a core from the entire length of the package, such samples of fertilizers and fertilizer materials as he may be directed by —— to inspect or that he may find uninspected; and in the performance of his duty he shall carefully draw samples as follows: In lots of ten packages or less, from every package; in lots of 10 to 100 packages, from not less than 10 packages; in lots of 100 packages and over, from not less than 10 per cent of the entire number; and, after thoroughly mixing the samples so drawn, he shall, by the method known as "quartering," draw from such thoroughly-mixed sample two subsamples, and with them fill two sample bottles, and shall plainly write on a label on said bottles the number of said sample, and shall also write on the label on one only of said bottles the name of the fertilizer, acid phosphate, or other fertilizer material, also the name of the manufacturers. He shall then seal both of the said bottles, and shall forward to —— the said samples so drawn by him, stating the number of sacks from which the sample was drawn, and a full report of the inspection written on a form prescribed by ——, which report must be numbered to agree with the number on the bottle; and in said report shall be given the name of the fertilizer or fertilizer material, the name of the manufacturer, the guaranteed analysis, the place where inspected, the date of the inspection, and the name of the inspector; and it shall be the duty of said inspectors to keep a complete record of all inspections made by them on forms prescribed by ——. Before entering upon the discharge of their duties they shall also take and subscribe, before some officer authorized to administer the same, an oath to faithfully discharge all the duties which may be required of them in pursuance of this act.

SEC. 11. Be it further enacted, That a sample of all fertilizers or fertilizer materials, drawn by the official inspectors and filed with the ——, shall be marked by number and delivered by said —— to the State chemist, who shall make a complete analysis of the same and certify, under same number as marked, said analysis to said ——, which analysis shall be recorded as official and entered opposite the brand of fertilizer or fertilizer material which the mark and number represent; and only the said official analysis of such fertilizer or fertilizer material, under the seal of ——, shall be admissible as evidence in any of the courts of this State on the trial of any issue involving the merits of such fertilizer or fertilizer material.

SEC. 12. Be it further enacted, That the —— shall have authority to establish such rules and regulations in regard to the inspection, analysis, and sale of fertilizers and fertilizer materials as shall not be inconsistent with the provisions of this act, and as in —— judgment will best carry out the requirements thereof.

SEC. 13. Be it further enacted, That nothing in this act shall be construed to restrict or avoid sales of acid phosphate or any other fertilizer material to each other by importers, manufacturers, or manipulators who mix fertilizer materials for sale, or as preventing the free and unrestricted shipments of material to manufacturers or manipulators who have registered their brands as required by the provisions of this act.

SEC. 14. Be it further enacted, That any person selling or offering for sale any fertilizer or fertilizer material without having first complied with the provisions of this act shall be guilty of a misdemeanor, and on conviction thereof shall be punished as prescribed in ——.

SEC. 15. Be it further enacted, by the authority aforesaid, That all laws and parts of laws in conflict with this act be, and the same are, hereby repealed.

COLLEGE PARK, MD., *November 14, 1903.*

DEAR SIR: In reply to your letter of October 29 I regret to say that I have been so busy with my various duties that I have not had time to give it the attention its importance deserves. The drafting of a State law which will be satisfactory is a difficult matter, but, I think, one to be general in its application would be a much more difficult task, due to the various conditions and different laws already in force in a number of the States. I should think a national law would, of necessity, have to allow considerable latitude in guaranties.

If we could all come to some reasonably close agreement in regard to guaranteed analysis, and that should be embodied in a national law, it would help matters considerably. Since hearing the views of the members of the committee some time ago I am about convinced that this is a hopeless undertaking. I am more fully convinced than ever of the necessity for simplicity in guaranties. Many of our farmers know nothing more than "a ton of phosphate" yet, and have no conception as to any relation between analysis and value. The statement of the phosphoric acid as "soluble" and "available" and "total," and each of these restated in the equivalent in "bone phosphate," and so on with the potash and the nitrogen, is not intelligible to one in a hundred of our farmers, and I doubt if one out of ten of the members of the working force of the agricultural colleges and experiment stations would fully understand it. It is all very well for chemists who have been connected with fertilizer inspection for years to talk about "total nitrogen," "organic nitrogen," "ammoniacal nitrogen," and "nitrate nitrogen," but it is too much for the average farmer—at least in Maryland.

However, I think that national legislation is desirable. The work should be under the Bureau of Chemistry. The repetition of guaranty in different forms and the giving of maximum analyses should, so far as possible, be prohibited. The expense should be defrayed by the Government. The legislation should follow, as nearly as may be, the general trend of inspection already established by the Bureau.

As to just how national legislation should influence State legislation I am not now prepared to state. There are questions of constitutional law, etc., involved, which can best be solved by those who have had considerable experience with them.

Very respectfully,

H. B. McDonnell, *State Chemist.*

St. Louis, Mo., *November 2, 1903.*

My Dear Sir: Your favor of October 29 has been forwarded to me from New York. In reply I would say:

(1) I believe that some legislation of this kind is desirable.

(2) An act which will provide (*a*) the purchaser a minimum guaranty in the plainest terms; (*b*) a revenue great enough to permit of the employment of high-class inspectors and chemists; (*c*) a method of raising this revenue that will be equitable and at the same time tend to prevent unnecessary multiplication of brands; a permanent registration fee (not annual) and a moderate tonnage tax will cover these points; the original registration fee should not be great enough to discourage small manufacturers from registering goods and furnishing local competition; this is especially important in the country west of the Mississippi River, and hence in a law relating to territories; (*d*) that the possessor of the goods as well as the vendor shall be held responsible for obedience to the provisions of the law; (*e*) a model on which local legislation may be based; (*f*) penalties of such kind and extent as to enforce the guaranty and prevent dishonest methods of competition among manufacturers; (*g*) a board of appeal for final decision of differences arising between manufacturers and official inspection bureau; (*h*) flexibility enough to meet new conditions that may arise in the trade.

(3). I believe that there should be no restrictions in regard to the amount of the three essential plant foods in any fertilizer, but the minimum amounts of each should be clearly and definitely stated. National legislation should encourage uniformity in State legislation:

In my opinion the "restriction of commerce" complaint is somewhat overworked. No one questions the fact that some labor and expense are required to conform to the present fertilizer laws. But these laws are the necessary consequences of frauds committed by parties engaged in the fertilizer industry. Fertilizer manufacturers make a fixed charge for "State tax and inspection" and in the cases I have investigated this fixed charge is so much greater than the actual cost that what is an apparent expense is converted into a source of profit. I am aware that provision *d*, holding the possessor, who may also be the consumer, responsible for the obedience to the law, is rather unusual and at present exists in only one State, Indiana. In practice it works admirably; the consumer makes the local agent protect him and the local agent in turn make his principal, the manufacturer, insure him (the local agent) against all costs and penalties arising from failure to comply with the law. In this way a State law is made to reach a manufacturer who is located outside the State. Direct shipments to consumers from outside States are thus reached by a simple provision in the contract, and as the goods are sold on credit, the purchaser is able to deduct from his payments any penalties he has suffered through failure of the manufacturer to fulfill his contract.

While giving due consideration to the somewhat hackneyed complaints about unnecessary restrictions of commerce, we must not lose sight of the causes that gave rise to these restrictions and of the tremendous financial and educational advantages which fertilizer consumers have derived from them. Nor must we lose sight of the fact that an efficient fertilizer law is the best protection that an honest manufacturer can have against unfair competition.

H. A. Huston,
Chemist, German Kali Works.

Auburn, Ala., *November 10, 1903.*

Dear Sir: Your letter of recent date relative to national fertilizer legislation came promptly to hand, and I have carefully noted the various points upon which you desire an expression of opinion by the members of your committee on fertilizer legislation.

With regard to the first point upon which you desire an expression, I would say that national legislation controlling interstate commerce in fertilizers in the District of Columbia and the several Territories would prove quite beneficial, in my opinion, and I also think it highly desirable, sooner or later, to enact legislation which will better facilitate the carrying on of trade in fertilizers between the several States.

From my connection for a number of years with the fertilizer control in this State, I have become aware of the hindrances which have been placed in the way of legitimate traffic and trade in fertilizers by reason of the diverse restrictions and regulations placed upon the traffic in different States. I would say further that as a result of a movement inaugurated by the Association of Commissioners of Agriculture of the Cotton States (including in its membership the official chemists of those States) that practically a uniform law has been adopted in a number of those States, while legislation along the same lines is pending in some of the remaining States. In North Carolina, Tennessee, Georgia, and Alabama (controlling a consumption of more than one and a quarter million tons) a uniform system of branding packages and of stating guaranties is now in force, thereby removing unnecessary and annoying restrictions upon commerce in fertilizers in those States.

With regard to the second question presented for the consideration of the members of the committee, I would say that I am hardly prepared to make definite suggestions as to the exact scope and character of an act which would accomplish the purposes in view.

So far as legislation for the District of Columbia and the Territories is concerned, it seems to me that the essential features of the scheme for fertilizer legislation,[a] proposed by this committee in 1897, could be embodied in the act to advantage.

Among some of the desirable ends to be accomplished by national legislation relating to interstate commerce in fertilizers, may be mentioned the securing of uniform systems of branding and of stating guaranties where shipments are to be made from one State to another; the prevention of the duplication of the same brand or trade name, and the protection of manufacturers having prior claims to the right of use of a given trade name; the prevention of the shipment of condemned or rejected goods from one State to another unless the goods are so branded as to show their true composition or fertilizing value, etc.

National legislation upon these lines would in a short time tend to bring about uniformity in State legislation relative to fertilizer traffic within the individual States, as no State would care to have two different systems for intrastate and interstate commerce.

Yours, very truly,

B. B. Ross,
State Chemist.

The members of the committee seem to be of the opinion that national fertilizer legislation is as much to be desired as is legislation in regard to commerce in foods, for the double purpose of protecting the honest manufacturer and the consumer against the mercenary manufacturer, and especially to unify the provisions of State legislation. The manufacturers and dealers in fertilizers are confronted by exactly the same problem as confronts the manufacturers and distributors of foods. Almost every State in the Union has a food law and a fertilizer law, and no two of these laws were identical, or even harmonious, until recently, when some efforts were made in this direction by several of the States. The result is that the same [illegible] e fertilizers must be put up and labeled one way in one State and another way in another State. This entails an increased amount of labor on the manufacturer, and the consumer has to pay for it, so the final result is simply to raise the price of fertilizer materials. Your committee is of the opinion that legislation of a national character regulating interstate and foreign commerce in fertilizers and fertilizing materials would be of great benefit in this respect.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Circular No. 3.

I also have the report of the committee on fertilizer legislation of the Association of Agricultural Colleges and Experiment Stations, which is submitted for publication. In the main the two committees are in entire harmony, although the purposes of the two are somewhat different.

REPORT OF THE STANDING COMMITTEE OF THE ASSOCIATION OF AMERICAN AGRICULTURAL COLLEGES AND EXPERIMENT STATIONS ON UNIFORM FERTILIZER AND FEEDING-STUFF LEGISLATION.

In the course of the past year your committee, as heretofore, has been in correspondence with parties in several States who were interested in the passage of new fertilizer laws or in the amendment of existing ones.

Arizona, Idaho, New Mexico, Nevada, Montana, Wyoming, and Utah have not yet felt the necessity of legislation in this line. In Colorado and Arkansas recent attempts to pass such laws have been defeated. The following reports have been received from some of the other States:

Ex-Director Huston, of Indiana, reports that the existence of the recommendations of this association was of much assistance in connection with steps taken to amend the old fertilizer law in that State. The law as enacted was made to correspond to the recommendations in certain particulars and the other points were practically all left to the discretion of the executive officer, thus rendering it possible to make rules in accordance with the recommendations.

Professor Ladd, of North Dakota, reports that at the last session of the legislature in that State a fertilizer law was enacted, and that the bill was drawn in accordance with the recommendations of this association, which he says were very helpful in the preparation of the bill "and in securing the necessary legislative action thereon."

R. E. Rose, State chemist, Tallahassee, Fla., writes that the law in that State has recently been amended to conform, in so far as possible, with the recommendations concerning uniformity. He adds that the recommendations were of material service.

Prof. F. B. Mumford, of Missouri, reports that the law in that State has been amended recently and that the recommendations were of "much assistance."

President McBryde, of Virginia, reported July 4, 1903, the changes in the law in that State were then being considered and that amendments in the line of the recommendations were being urged. In conclusion he says: "It follows, therefore, that your recommendations will be helpful in securing the legislation needed."

Director Armsby reports that the recently amended law of Pennsylvania conforms very largely in substance to the recommendations.

Director Soule, of Tennessee, states that a new law was passed in that State in April, 1903. The law was drawn with the object of making it conform with the recommendations of the associations, but a few amendments were made not in harmony therewith which it is believed weakened the law. He adds that it is hoped later to secure such amendments as will make the law conform to the original draft, and that "Had it not been for the existence of the recommendations, it would probably not have been possible to secure the passage of the present law."

Director J. F. Duggar, of Alabama, writes under date of July 7 that in that State "the old law has been replaced this year by a new one, which embodies the recommendations of the Association of American Agricultural Colleges and Experiment Stations and of the Association of Official Agricultural Chemists," and that "the recommendations alluded to have had much weight in securing the revision of legislation along this line."

After careful consideration of the subject, your committee submits the following recommendations regarding laws regulating the sale of feeding stuffs.

(1) That for the purpose of defraying the expenses of feeding-stuff inspection the State should make a direct appropriation, or where this is impracticable a brand tax should be levied. In view of the experience of Maine and Vermont a tonnage tax is not to be recommended.

(2) That the following materials should be exempt from the provisions of feeding-stuff laws; hays and straws and whole unmixed seeds, such as wheat, rye, barley,

oats, Indian corn, buckwheat, broom corn, peas, and the unmixed meals of the entire grains of such seeds.

(3) The term "concentrated feeding stuff" should include linseed meals, cotton-seed meals, cotton-seed feeds, pea meals, cocoanut meals, gluten meals, gluten feeds, maize feeds, starch feeds, sugar feeds, dried brewers' grains, dried distillers' grains, malt sprouts, hominy feeds, cerealine feeds, germ feeds, rice meals, oat feeds, corn and oat chops, corn and oat feeds, corn bran, ground beef or fish scraps, condimental foods, poultry foods, stock foods, patented proprietary or trade-mark stock and poultry foods, and all other materials of a similar nature not included in section 2 above. Where practicable the by-products from the milling of wheat, rye, and buckwheat should be included under the requirements of the laws.

(4) That a legible printed statement should be affixed to or printed on each package containing a feeding stuff named in section 3, giving the net weight of the package, the name and address of the manufacturer or importer, the name, brand, or trade-mark under which the article is sold, and the guaranteed analysis showing the percentage of crude protein and of crude fat and a maximum of fiber which shall not be exceeded.

The law should provide that the chemical analysis, including the determinations of crude fiber, crude protein, and crude fat shall be made by the official methods of the Association of Official Agricultural Chemists.

If the feeding stuff is sold in bulk or put up in packages belonging to the purchaser, the agent or dealer shall furnish him with a certified statement of the net weight of the lot, the name and address of the manufacturer or importer, the brand or trade-mark under which said article was sold, and the percentage of crude protein and crude fat which said article is guaranteed to contain, as determined by the official methods of the Association of Official Agricultural Chemists.

(5) That a certified copy of the statement in section 4 above be filed with the executive officer each year.

(6) That the law should contain a penalty by fines only for violations of its provisions.

The committee recommends to the Association of American Agricultural Colleges and Experiment Stations the adoption of the recommendations 1 to 6, inclusive, with the suggestion that this or some other committee should be instructed to use its efforts to secure the end in view by using its influence to aid in securing uniform legislation in the several States.

Respectfully submitted.

H. J. Wheeler.
Chas. D. Woods.
E. H. Jenkins.
H. P. Armsby.
M. A. Scovell.

I would like also to call attention to the suggestion which was made to me by Mr. W. S. Myers, director of the nitrate of soda propaganda in this country. Under date of November 12, he writes as follows:

Dear Sir: It has occurred to me, as doubtless it has to you, that the practice of our experiment stations in reporting available phosphoric acid and potash, and failing to report available nitrogen, does not disclose to the consumer the value of the fertilizer from either a commercial or a merely agricultural standpoint. The official methods now in use are adequate at least to make a preliminary classification of the nitrogenous content of fertilizers, but the usual practice of the station laboratories is to determine and report total nitrogen only. Is this not most unfortunate for the farmer, for whether the phosphoric acid is worth 2 cents or 4 cents a pound does not seem as of great moment as whether the nitrogen is worth 4 cents or 16 cents a pound? Even though the nitrogenous content is small in the average commercial fertilizer, small percentages at high prices cut a large figure in valuations.

The purpose of this note is to ask the Association of Official Agricultural Chemists to consider recommending by resolution the practice of putting into use the methods already adopted for nitrogenous fertilizers. At the present time most of the stations are not willing to take the trouble. I am aware that methods are under investigation for the classification of organic nitrogen. Does not this seem rather premature in view of the failure to put into practice the simple methods already adopted? To put the case concretely, from my standpoint, the determination of nitrates of ammonia salts and of organic nitrogen should be made in each fertilizer whenever found.

Very sincerely, yours,

William S. Myers.

It appears from the views expressed by the members of the committee and others that the general opinion is that some national legislation should be enacted, but there is no definite expression as yet by the committee as to the form that this legislation should take. I therefore move that the committee on fertilizer legislation be instructed to confer with manufacturers and distributers and with such other persons as may be interested in the matter and report at the next meeting the form that national legislation should take with a view to preparing a tentative bill and urging its enactment into a law.

The motion was adopted.

Mr. WILEY. I would like to offer two resolutions. The first one pertains to the advisibility of associating the National Bureau of Standards with the work of this association. I have had a conference with the chief chemist of the Bureau of Standards, and, while he would not commit himself on account of his inability at that time to consult with the director of the Bureau, he looked very favorably upon the proposition which I made, to the effect that, as the Bureau of Standards was now established by law as the final court of resort for all matters relating to standards, it would be desirable for us to collaborate on standard methods of analysis.

It can do no harm, therefore, to express our desire for such collaboration if it can be secured after consultation with the officials of that Bureau. I therefore move the following resolution:

Resolved, That the Bureau of Standards be invited to cooperate with the referees of the association in so far as possible in establishing the accuracy of standard methods of analysis.

Adopted.

[The following letter has been received from Director Stratton, and the referees are therefore requested to ask the Bureau of Standards to collaborate in their work. H. W. W.]

DECEMBER 8, 1903.

DEAR SIR: I beg to acknowledge the receipt of your letter of December 4, transmitting resolutions passed by the Association of Official Agricultural Chemists inviting the Bureau of Standards to cooperate with the association in establishing the accuracy of standard methods of analysis. In reply I would state that the Bureau of Standards will be pleased to cooperate with the association in every way consistent with its functions. This question is one in which the Bureau, and especially our chemical section, is very much interested.

Respectfully, S. W. STRATTON, *Director*.

Mr. WILEY. The second resolution which I propose to offer relates to a very important part of the work of agricultural chemistry recently taken up by the Department of Agriculture, which has realized the importance to agriculture of the cultivation of medicinal plants. Agriculture, of course, includes forestry and horticulture, and the territory of the United States is now so extended that within the jurisdiction of the United States almost every plant which is used in medicine is grown. Hence the action of Congress in establishing two different forms of investigation of medicinal plants in the Department of Agriculture—one relating particularly to the botanical side of the work in the discovery and introduction of new and useful medicinal plants within the jurisdiction of the United States, and the other in establishing a drug laboratory where such plants can be analyzed and their medicinal properties determined. Inasmuch, therefore, as that work has been recognized by Congress as a part of the official work of the Department of Agriculture I move the following resolution:

Resolved, That a referee on the analysis of medicinal plants and drugs made therefrom be appointed by this association.

Adopted.

Mr. VAN SLYKE. If there is no objection, I would like to offer two resolutions. The first one relates to a request to be made of the secretary in regard to printing the recommendations that are passed by the association. According to our constitution a recommendation which carries with it a change of method can not be acted upon finally by the association until the members have had an opportunity to test the method as modified. That, as matters now stand, practically involves a postponement of a year, or, practically, two years. If we can place in the hands of the members within a comparatively short length of time after the adjournment of each meeting the results of the association's work in the form of recommendations, there will be an opportunity to test these methods, and the time of adoption will be hastened. I therefore move the following resolution:

Resolved, That the secretary be requested to print the changes in methods and instructions recommended by the association and distribute them to the members at as early a date as is practicable.

Adopted.

Mr. VAN SLYKE. The other resolution I have in mind relates to the place of meeting for next year. The constitution provides the association shall determine the place of meeting. I move that the executive committee be authorized to select a place of meeting next year and that they be requested to consider the feasibility of having that meeting in St. Louis.

The executive committee already has the power to determine the time. The association, unless it delegates the power to the committee, determines the place.

Mr. HUSTON. As a resident of the city of St. Louis I feel sure you would receive a very cordial welcome if you should meet in that city. There will, of course, be many congresses there next year, and our meetings, if arranged to accord with some others, would, I think, be doubly interesting. The Association of American Fertilizer Manufacturers have arranged to meet there, and I am sure the meeting will be unusually interesting to the members of this association, as it will be of a scientific nature.

Mr. **Wiley**. We have a good precedent for this action in the two meetings that have been held outside of Washington. All who were present remember with pleasure the Chicago meeting in 1903, and the San Francisco meeting in 1899, which was a far greater success in point of attendance than anyone had anticipated. I think we are rather tardy in honoring the man who did so much to make that meeting a success—our president-elect, Mr. Jaffa. I am heartily in favor of the suggestion made, and am sure the executive committee will be glad to consider the suggestion to arrange for the next meeting in St. Louis.

The motion of Mr. Van Slyke was carried.

Mr. **Coates**. Have any steps been taken to secure the revision of Bulletin No. 46?

Mr. **Wiley**. We have ready for mortising or resetting many of the changes that have been made since Bulletin No. 46 was printed. By reason of the expense of resetting, it is deemed wise to wait until the methods had settled into more definite form. You all know how rapidly the methods were changed and how slowly, fortunately, they are now changed. We now hope to issue an entirely new edition of Bulletin No. 46 very soon.

The **President**. Is it the intention to embody in the revised edition the methods as published in Bulletin No. 65 for the analysis of foods and food products?

Mr. **Wiley**. We might do that. Some of these methods, however, are only offered for trial and could hardly be made official or even provisional.

Mr. **Bigelow**. It might be well to have an expression of opinion on this point by the association, as to whether it would be desirable to print all the methods together. We have now several classes of chemists in the association; many of the experiment station chemists have no interest in the food methods, except perhaps to have them accessible, and others, belonging to the boards of health and dairy and food commissions, have no interest in the other methods, such as those on soils and fertilizers. If the two lines of work are published together it would make a bulletin of about 250 pages. At the same time there are some who are interested in both lines of work. Another point is that the food methods will change radically and in detail for some time to come. It is probable that they will not be in a shape to be adopted by the association finally for several years.

The **President**. Considering the remarks of Mr. Bigelow it seems advisable in the case of the present revision, at least, to print the food methods separately. I would like to have an expression of opinion from other members of the association.

Mr. **Morse**. I should prefer to have them separate. It is more convenient on account of the size, and as changes are sure to be made in the food methods it would take longer to get these into print if they are all printed together.

Mr. **McDonnell.** It seems to me it should be left largely in the hands of the Secretary. From what has been said it appears, however, to be more convenient and better in every way to have two bulletins.

Mr. **Williams.** What action is going to be taken, if any, with reference to printing the Dancy and Battle conversion tables?

Mr. **Wiley.** If these tables are offered as a part of the proceedings, they will be published as such. I think they were offered by the authors, without any interest in the copyright, for the use of the association.

Mr. **Williams.** They only asked credit for their work.

Mr. **McDonnell.** I have no objection to printing these tables, but I think those printed by the Maryland station, which I have submitted, are more compact and convenient, and it might be well for the association to compare the two before printing.

Mr. **Wiley.** As the constants of nature are constantly changing it might be well, if the association does ask for the publication of tables which have been calculated for a number of years, to recognize the fact that a number of new elements have been discovered during that time and some of the old ones have been reduced in dignity and some increased, and to have a careful revision of the calculations before printing them.

The **President.** I would suggest that a committee be appointed, authorized to prepare either from the Dancy and Battle tables or Mr. McDonnell's bulletin tables for publication in the Proceedings.

Mr. **Hardin.** Down South the chemists still use the Dancy tables for nitrogen and phosphoric acid. The potash tables we have not been able to use for several years, as a new factor has been adopted. Most of us prefer the form of these tables, and I think myself that they are the best that have ever been published. But in getting them up again they would have to be revised.

Mr. **Huston.** Perhaps it would not be well to print any of these right away. There is an excellent set of tables in use in the Indiana laboratory, calculated by Mr. Jones, which brings the potash up to the present weight, and the table for nitrogen is exceedingly useful, as it is based on the official methods in use. A committee to decide what tables should be published would be very useful. I am quite sure that the Indiana tables could be used, either in whole or in part, if acceptable to the committee.

Mr. **Wiley.** I move that the president be requested to appoint a committee of three, of which Mr. H. B. McDonnell shall be the chairman, to consider the matter of publishing in the Proceedings of the association calculation tables, and that the committee be authorized to submit its report to the secretary of the association with recommendation.[a]

Adopted.

Mr. Wiley. I would like to call the attention of the association to the fact that this is a record-making meeting in the point of attendance. Over 140 members have registered during the convention. This shows what a vigorous growth the association is now having. I would like to say a word as one of the Nestors of the association, with a record which no one else has, of having been present at every meeting since its organization. What we need now particularly in this association is the more vigorous cultivation of skill in analysis. Altogether too few of our members take part in the referees' work.

There are two benefits which accrue to every member of the association taking part in the work of the referees; one is the benefit he confers upon the association, and the other the benefit he will confer upon himself, because if he be in any way inaccurate, or if he fail to reach the standard of excellence of this association, he will be certain to find it out the moment he undertakes collaborative work and be able to correct any fault he may have.

In the establishment of standards and the securing of data for outlining methods accuracy is absolutely indispensable. The formation of theories is not a difficult thing if you have the right basis; and I recall especially the remarks made by Professor Crookes, in his address before the Berlin congress last June, that no theory of any kind is of any value unless every fact which is to be correlated in that theory finds a place there. And so it is that in the great discussions of the problems relating to agricultural chemistry we must be sure first of all of the ground on which we stand.

As I look over this association I realize, what we see everywhere, that it is the young men who rule the world. We old chaps are altogether in the minority. Presumably it is the young men who need the training most. While they are just out of college and have done a certain amount of practical work, they have not had the necessary training for such investigations, and I especially appeal to the young men—and to the young women too, because we are seeing evidences of the other sex breaking over into our ranks—I earnestly appeal to them to take an active part in the work of the referees. I would not have a chemist in my Bureau who would not take part in this referee work. I not only require it but I give every opportunity for doing such work. I would say to the trustees of every station, "Send your chemists to the meetings." As a matter of fact we do have the greater part of these stations represented, but they ought all to be represented, and the station chemists should be afforded every facility to engage in collaborative work. The Secretary of Agriculture affords every facility to the chemists in the Department of Agriculture and encourages them to engage in the work of this association. Long before it was made by Congress his advisor he did that, and of course he is still more interested now. We are the only association of this kind that has any official standing by act of Congress, and therefore we are under special obligations to the people of this country to perfect ourselves in everything that lies at the foundation of our science. So, let us have more collaboration, especially among the younger members of the association, and instead of two or three people reporting results, let us have thirteen or fourteen, even if four-fifths of them have to be sent back as not fit for publication. [Applause.]

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[a] The report of this committee is given on page 220.

The PRESIDENT. I appreciate very much what Doctor Wiley has said, and he has emphasized certain remarks that I made in the opening address. It is very regrettable that our referees have so little cooperation in their work. Every individual of this association ought to make it his business to do some work on one of the subjects under consideration, and I think the time is coming when those who do not will be considered as deadwood in the association.

I also wish to call special attention to another point which I think has been mentioned in regard to the sugar work. This is the graduation of the volumetric apparatus at too low a temperature, 15.5°. I think something should be done toward having the graduation made at some temperature more suitable to that of our laboratories.

Is the committee on resolutions ready to report?

Mr. McDONNELL. In the absence of the chairman of this committee, I move that the secretary extend to the officers of Columbian University, the Cosmos Club, and to the Secretary of Agriculture our thanks for the courtesies extended to the association.

Adopted.

Mr. BIGELOW. Mr. Walter Bernays has asked me to call the attention of the association to the fact that no effort has been made as yet to establish methods for the examination for disinfectants, and it is a subject with which the boards of health are having much trouble, and they would like to have it taken up. It is a question whether this subject would come within the province of the association, and some expression of opinion is desired as to whether it would be desirable to consider it.

Mr. COATES. A few years ago there was a yellow fever scare in a town in Louisiana, and the place was flooded with bleaching powder. I analyzed some of it and found it contained less than 2 per cent of available chlorin, and could not find any specimen in town which contained more than that. This question is going to be more and more important as time goes on, and I would like very much to see Mr. Bigelow's suggestion acted upon.

Mr. BIGELOW. I move that a referee be appointed to take up this question.

Mr. HAYWOOD. As we have a number of reporters now and have just created a new one on drugs, it seems hardly necessary to appoint a referee for this special work. The work would be very much along the line of our present investigations on insecticides. I think it would be better to refer this subject to the referee on insecticides, and therefore offer an amendment to the effect that the establishment of methods for the analysis of disinfectants be referred to the referee on insecticides and fungicides.

Mr. BIGELOW. I accept the amendment.

The motion as amended was passed.

The PRESIDENT. Is the chairman of Committee A on recommendations, ready to report?

REPORT OF COMMITTEE A ON RECOMMENDATIONS.

B. L. HARTWELL, *Chairman.*

(Phosphoric acid, potash, nitrogen, soils, ash, and insecticides.)

1. PHOSPHORIC ACID.

No recommendations were made by the referee, and those made by the associate are practically covered by the recommendations on soils.

2. POTASH.

It is recommended—

1. That the question of the further trial of the modification requiring a slightly acid solution and neutralization with sodium hydroxid be left to the discretion of the referee. (Adopted.)
2. That the association take some action to ascertain whether the members desire to determine moisture in potash salts by drying at a stated temperature or by ignition, and that the study of the method thought most desirable be continued with a view to obtaining more concordant results for water in potash salts.

(The committee, without action, presented paragraph 2 to the association, which referred the whole question to the referee for 1904.)

On motion by Mr. Ross, the association adjourned at 12.50 until 2 o'clock, the report of Committee A to be completed at the afternoon session.

SATURDAY—AFTERNOON SESSION.

The PRESIDENT. On the committee to report to the secretary on calculation tables, of which Mr. McDonnell is the chairman, I will appoint Mr. Williams and Mr. Ross.

REPORT OF THE COMMITTEE ON CONVERSION TABLES.

[The following letter was submitted to the secretary of the association, in accordance with the resolution passed, together with the tables selected. It was not clear, however, from the instructions given, whether the association desired the secretary to take action on the report of the committee and publish the tables recommended or merely to receive the report; and further, there seemed to be a difference of opinion as to whether the tables were to be printed in the revised edition of Bulletin 46, on Methods of Analysis, or in the Proceedings. In view of these facts the secretary submits the report herewith and holds the tables recommended for the action of the association at the meeting of 1904. H. W. W.]

College Park, Md., *February 17, 1904.*

Dear Sir: On behalf of the committee appointed by the Association of Official Agricultural Chemists, consisting of Mr. B. B. Ross, Mr. C. B. Williams, and myself, to select conversion tables for publication in the Methods of Analysis, I beg to report that a majority of the committee have decided to recommend McDonnell's tables, but think that in view of the fact that the atomic weights have been revised since the tables were first published, making slight changes in the factors, they should be recalculated and a protein table added. This I have done with the assistance of Messrs. R. H. Kerr and William R. Wharton. The revised tables are sent herewith.

Very respectfully,

H. B. McDonnell,
Chairman Committee on Conversion Tables.

Mr. Winton. Referring to Mr. Hillebrand's report of this morning I wish to move the following resolution:

Resolved, That the Chair be instructed to appoint a committee from this association to cooperate with the committee of the American Chemical Society in the matter of testing chemicals for purity.

Adopted.

The President. I will appoint Mr. Kebler, Mr. Winton, and Mr. Kilgore on this committee.

The report on tannin will now be received.

REPORT ON TANNIN.

By G. A. Kerr, *Referee.*

Pursuant to the recommendations made in 1902, the referee sent to 16 chemists samples of fluid chestnut and solid quebracho extracts for the purpose of—

(1) Confirming the use of a single filter-paper in determining soluble solids.

(2) Investigating the Proctor correction for filter paper-absorption, as adopted by the I. A. L. T. C.

(3) Ascertaining the effect, if any, of using a specified dilution in comparison with the variation now allowed by the official method.

Samples of acidified hemlock liquor were also sent out, for the purpose of further testing the present provisional method, and also an experimental method, suggested by W. H. Teas, for the determination of acids in tanning liquors.

An effort was made by the referee to procure a dry chromed hide powder for comparison with the present wet hide powder method, but this effort was unsuccessful owing to the impossibility of procuring in time for this year's work, a hide powder which could be dried with the retention of its absorptive qualities.

Study I.

Instructions were given to determine the total solids, soluble solids, and insolubles in samples No. 1 and No. 2, by the association method.[a] The results returned are contained in Table I.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Circular No. 8, revised.

TABLE I.—*Determinations of total and soluble solids and insolubles by the provisional method.*

No. of determination.	Chestnut extract (A).			Quebracho extract (B).		
	Total solids.	Soluble solids.	Insolubles.	Total solids.	Soluble solids.	Insolubles.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	37.16	36.18	0.98	86.61	79.56	7.05
2	37.43	36.40	1.03	87.61	81.55	6.06
3	37.62	36.92	.70	88.49	82.00	6.49
4	37.01	36.15	.86	86.99	81.80	5.19
5	37.78	36.69	1.09	87.50	82.30	5.28
6	37.42	36.35	1.07	86.76	80.48	6.28
7	37.20	36.14	1.06	86.55	79.82	6.73
8	37.73	36.95	.78	88.72	81.12	7.60
9	37.69	36.43	1.26	88.55	81.57	6.98
10	36.37	35.16	1.21	86.35	80.28	6.07
11	38.37	36.88	1.49	85.09	78.91	6.18
Average	37.43	36.38	1.04	87.20	80.85	6.35
Maximum	38.37	36.95	1.49	88.72	82.30	7.60
Minimum	36.37	35.16	.70	85.09	78.91	5.19
Greatest difference	2.00	1.79	.79	3.63	3.39	2.41
Greatest variation from average	1.06	1.22	.44	2.11	1.94	1.24

In the soluble solids column, chestnut extract (A), the greatest difference from the average quantity of soluble solids found by 11 analysts is 3.36 per cent of the whole. In the quebracho extract (B) it is 2.39 per cent. While these figures confirm last year's work with reference to the adoption of the single-filter paper for filtration of soluble solids, they do not convey an adequate idea of the variation between the highest and lowest percentage of soluble solids found by the different analysts.

Under A the greatest variation is 1.79 per cent, and under B, 3.39 per cent. When it is considered that these differences have a direct bearing upon the tannin figure, the weakness of the present method of determining soluble solids is very apparent.

An examination of the columns giving total solids and insolubles reveals a condition equally unsatisfactory, and makes the seat of trouble somewhat uncertain, commencing as it does with a variation of over 5 per cent and 4 per cent in the total solids columns, and ending with one of 112 per cent and 37 per cent in the insoluble columns, the indication would seem to be that the entire method, as applied to these items, needs revision.

From personal observation and the comments of the collaborators in this work the referee is of the opinion, however, that the method is not entirely accountable for the differences in results, as it has been demonstrated that with close attention to detail of manipulation and operation much more concordant figures than the above can be obtained. Attention is especially called to the necessity for protecting fluid extracts against loss by evaporation while weighing same for dilution; this also applies to the absorption of moisture by pulverized dry extracts.

The drying of residues, and the loss from evaporation that ensues during the filtration of soluble solids, also require closer attention. The error from the latter cause is much greater, apparently, than has been supposed, and the referee would recommend that as a specific means of guarding against it the following be embodied in Paragraph IV, Soluble solids,[a] of the method: "To guard against loss by evaporation, flasks receiving filtered liquor shall be stoppered with a perforated rubber stopper, and the funnel stem inserted through perforation. Funnels shall be covered during filtration with a glass plate, having an orifice only sufficiently large to admit air required by displacement of liquor."

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[a] U. S. Dept. Agr., Bureau of Chemistry, Circular No. 8, revised.

It is further recommended that the last sentence of Paragraph II, Quantity of material,[a] be changed to read: "In the case of extract weigh in weighing bottle fitted with ground-glass stopper, such quantity, etc."

Mr. Veitch makes the following comment:

> I have never been satisfied that the change from the double filter to a single one, was a wise one, owing to the fact that kaolin is so apt to run through a single paper. It has usually been my experience, even after rejecting the prescribed volume of liquor, that some kaolin still passes through, and may be best seen by carefully tilting the flask in which the filtrate is received.

STUDY II.

This experiment consists of a trial of the filter paper absorption correction as suggested by Mr. Proctor and adopted by the I. A. L. T. C. The following instructions are copied verbatim from the official method of the above association, except that the method of filtration was carried out according to the method of this association.

> (*a*) To determine the correction, filter clear about 500 cc of the tannin solution, of the strength prescribed for analysis. After thoroughly mixing, 50 cc is to be evaporated, to determine soluble solids (1). A portion of the remainder is then to be filtered in precisely the same manner as for (1), and 50 cc of the filtrate is to be evaporated for soluble solids (2).
>
> (*b*) Deduct (2) from (1) and the difference found is the correction which must be added to the soluble solids (1) to give the corrected soluble solids.
>
> (*c*) In proceeding with these filtrations use 2 grams (weighed) of kaolin, which is first to be washed with 75 cc of the liquor by decantation and then washed onto the filter with a further quantity of liquor; reject the first 150 cc which passes through in each filtration. Keep the filter full and guard against evaporation.

The results of this study are given in Table II.

TABLE II.—*Determination of correction for soluble solids.*

Determinations.	Chestnut extract.					Quebracho extract.				
	Total solids.	Soluble solids (1).	Soluble solids (2).	Difference.	Corrected soluble solids.	Total solids.	Soluble solids (1).	Soluble solids (2).	Difference.	Corrected soluble solids.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
1	37.35	36.35	36.19	0.16	36.51	86.92	79.67	79.23	0.44	80.11
2	37.43	36.45	36.14	.32	36.77	87.61	81.66	80.49	1.17	82.83
3	37.63	37.33	37.06	.27	37.60	88.52	84.25	83.48	.77	85.02
4	36.37	35.32	34.94	.38	35.70	86.35	80.34	79.89	.45	80.79
5	36.95	36.15	35.63	.52	36.67	87.05	81.85	80.49	1.36	83.21
6	37.78	36.69	36.44	.25	36.94	87.58	82.30	81.95	.35	82.65
7	37.42	36.63	36.52	.11	36.74	....	81.30	80.55	.75	82.06
8	37.37	36.73	36.48	.25	36.98	86.74	81.01	80.14	.87	81.88
9	37.45	36.73	36.70	.03	36.76	88.39	81.72	80.50	1.22	82.94
10	37.64	36.85	36.48	.37	37.22	88.32	83.40	82.51	.89	84.29
11	....	37.17	36.82	.35	37.52	....	80.30	80.15	.15	80.45
12	....	37.06	36.02	1.04	38.10	.....	77.13	76.93	.20	77.33
Average	37.33	36.62	36.28	.33	36.95	87.497	81.24	80.52	.718	81.96
Maximum	37.78	37.33	37.06	1.04	38.10	88.52	84.25	83.48	1.36	85.02
Minimum	36.37	35.32	34.94	.03	35.70	86.35	77.13	76.93	.15	77.33
Greatest difference	1.41	2.01	2.12	1.01	2.40	2.17	7.12	6.55	1.21	7.69
Greatest variation from average	.969	1.30	1.34	.70	1.259	1.47	4.11	3.59	.64	4.63

The foregoing results indicate the necessity of having ultimately a correction unless a nonabsorbent filter paper is secured or the filter paper now in use can be thoroughly tanned by the liquor passing through before the portion necessary for evaporation is taken. At present, however, it is plain, especially in the case of quebracho extract in Table II, that the method employed to ascertain this correction augments to an exaggerated extent the error already existing in the determination of soluble solids, which has the same bearing on the tannin content as the correction is intended to have. In consideration of this condition the referee would suggest that until more uniform results are obtained on the items under discussion further experiments in this line be deferred.

In both cases the differences cover a wider range than in Table I, but with the quebracho extract in Table II the differences are abnormal. It seems to be the consensus of opinion that this difference is largely due to loss from evaporation during the extended length of time required to filter the necessary quantity of liquor for the determination. In this connection it is interesting to note that the precautions employed by the various analysts to guard against evaporation do not seem to have been very effective, which fact emphasizes the necessity of adopting specific means for its prevention, as suggested by the referee in discussing the first experiment. The analysts make the following comments:

Mr. Veitch.—From the results of studies 1 and 2 it appears that the absorptive power of the filter is not satisfied by filtering 150 cc of solution. This is particularly apparent in the case of quebracho.

Mr. Mosbaugh.—Just after Mr. Proctor published the method I gave it some attention, and obtained satisfactory results in all extracts with the exception of solid quebracho. Nevertheless I am in favor of its being adopted, as it is well known that filter paper absorbs more or less tannin and this method of correction apparently remedies the evil.

Mr. Reed.—Personally I am not in favor of the method.

Mr. Teas.—I think the evaporation, taking place during filtration, accounts for the high soluble solids.

STUDY III.

For the third study the following instructions were given; results will be found in Table III:

Using official samples Nos. 1 and 2 determine total solids, soluble solids, and insolubles in each according to the provisional method, except as to dilution.

(a) For dilution of sample No. 1 use exactly 1.6 grams per 100 cc.

(b) For dilution of sample No. 2 use exactly 0.6 grams per 100 cc.

Weigh out the portion of sample No. 1 for dilution, in a covered vessel, to guard against evaporation. It is preferable to weigh out approximately the quantity required and dilute so that each 100 cc will contain 1.6 grams of the extract.

It having been suggested to the referee that the range of variation in the quantity of tannin admissible in dilutions according to the association method was too great to give uniform results (there being a difference of over 28 per cent between maximum and minimum), this experiment was undertaken to determine, by comparison with Table I, the value of results obtained by using a specific quantity of total solids. Comparing these tables, we find the results to be in favor of the specified quantity test; slightly so in sample 1, Table III, but more markedly so in sample 2.

The improvement will, perhaps, scarcely justify any change in the official method for the present, but the referee would suggest that the experiment be repeated with varying quantities of extract until it is ascertained whether or not the total solids contained by an extract form a better basis for dilution than the assumed tannin content. As there is evidently some error in drying residues, caused by the varying quantity of total solids in different dilutions, it seems to the writer that it would be easier to adjust the quantity of residue to be dried to meet the present conditions governing drying than to alter the method of drying to suit the difference in quantity of residue obtained by the association method now in use, inasmuch as this item has been more thoroughly worked out than any other in tannin analysis, excepting non-tannins.

TABLE III.—*Determinations by association method with modifications as to dilution.*

Determinations.	Sample 1 (chestnut extract, 16 grams per liter).			Sample 2 (quebracho extract, 6 grams per liter).		
	Total solids.	Soluble solids.	Insolubles.	Total solids.	Soluble solids.	Insolubles.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	37.31	36.26	1.06	87.31	81.28	6.03
2	36.95	36.16	.79	86.75	79.61	7.14
3	36.22	35.23	.99	88.63	80.76	5.54
4	37.74	37.06	.68	88.35	81.41	6.94
5	37.73	36.70	1.03	87.60	81.87	5.73
6	36.89	36.09	.80	86.50	80.51	5.99
7	37.25	36.00	1.25	86.83	80.66	6.17
8	37.30	36.27	1.03	86.80	79.87	6.93
9	37.63	36.49	1.14	87.58	80.56	7.02
10	37.65	36.87	.78	88.83	82.07	6.76
11	37.65	36.60	1.05	88.88	81.37	7.51
12	37.97	36.68	1.31	....	....	....
Average	37.36	36.36	.99	87.43	80.90	6.52
Maximum	37.97	37.06	1.31	88.88	82.07	7.51
Minimum	36.22	35.23	.68	86.30	79.61	5.44
Greatest difference	1.75	1.83	.63	2.58	2.46	2.07
Greatest variation from average	1.14	1.13	.31	1.45	1.29	1.08

STUDY IV.—ACIDITY OF LIQUORS.

For these experiments a sterilized neutral hemlock liquor (specific gravity 1.015), to which 0.15 per cent of acetic acid had been added, was sent out, and analysis requested by the following methods:

(*a*) By provisional method, paragraph 8, circular No. 8 revised, Bureau of Chemistry.

(*b*) To 100 cc of diluted liquor (prepared as in the provisional method) add 2.5 grams chemical pure animal charcoal, stir every fifteen minutes for two hours, filter, and titrate an aliquot portion with N/10 alkali. For results see Table IV.

TABLE IV.—*Total acidity of liquors as acetic acid.*

Determinations.	Provisional method.	Experimental method.
	Per cent.	*Per cent.*
1	0.195	0.161
2	.178	.137
3	.135	.150
4	.150	.156
5	.183	.149
6	.174	.140
7	.168	.144
8	.156	.120
9	.165	.105

TABLE IV.—*Total acidity of liquors as acetic acid*—Continued.

Determinations.	Provisional method.	Experimental method.
	Per cent.	*Per cent.*
10	0.170	0.120
11	.180	.120
12	.148	.140
Average	.1668	.1368
Maximum	.195	.161
Minimum	.135	.105
Greatest difference	.060	.056
Greatest variation from average	.0318	.0318

It will be noted that the provisional method gives results about 11 per cent higher than theory, and the experimental method the same amount lower.

By referring to Table IV in the referee's report for 1902[a] it will be seen that practically the same results were obtained. The percentage of variation from the average is very similar, although the liquor for 1902 evidently contained about twice as much acid as that sent out this year.

In the opinion of quite a number of analysts this provisional method of determining the acidity of liquors is very satisfactory in tannery practice, for which it is almost exclusively used, especially as to the concordance of the results obtained. Notwithstanding this the referee would suggest that further experiments be conducted with reference to the loss of acid presumably held or neutralized by the charcoal in method "b."

COMMENTS OF ANALYSTS.

Mr. Mosbaugh.—The acid determination I find does not cover the range of liquors found in sole-leather yards. While the results obtained are concordant and uniform, the method is not applicable to liquors higher in specific gravity than 1.030. As it is sometimes necessary to determine the acidity of the new as well as of the sapped liquors, it is impossible to get a filtrate sufficiently clear for titration, unless a weaker dilution is made or more charcoal used, and in either case the results are not as satisfactory as when the same amount of charcoal is used. I find that by carefully using gelatine in connection with charcoal very good results are obtained and approximately the correction as used for charcoal method holds out.

Mr. Stilwell.—In titrating liquor in Study IV I used for the alkali sodium hydrate, checking with limewater. I do not get good results when using N/10 soda, and of the three alkalis prefer limewater.

Good results are obtained by using 2 to 2.5 grams of charcoal and running in a shaker for from to two to three minutes. Decolorization is satisfactory. The best concentration is 0.2 to 0.25 grams of tannin per 100 cc of tan liquor.

In addition to the determinations prescribed in Study IV, Mr. Reed also determined the acidity with charcoal furnished by last year's referee (Mr. Teas), with the following results:

(*a*) Provisional method: 2.75 cc N/10 sodium hydrate to neutralise 40 cc of solution. Total acidity = 0.206 per cent as acetic acid.

(*b*) Cold charcoal method: 2.25 cc N/10 sodium hydrate to neutralise 40 cc of solution. Total acidity = 0.169 per cent as acetic acid.

Method (*a*) using 2.5 grams of charcoal furnished by this year's referee, instead of two grams as provided, gave a clearer filtrate in the former than in the latter case;

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[a] U. S. Dept. Agr., Bureau of Chemistry Bul. No. 73, p. 81.

2.3 cc N/10 sodium hydrate to neutralise 40 cc of solution. Total acidity = 0.173 per cent as acetic acid.

Method (*a*): 2 grams of charcoal = 0.195 per cent total acidity.
Method (*a*): 2.5 grams of charcoal = 0.173 per cent total acidity.
Loss due to 0.5 gram of charcoal = 0.22 per cent total acidity.
Loss due to 2.0 grams of charcoal = 0.88 per cent total acidity.
Corrected total acidity = 0.195 per cent + 0.088 per cent = 0.283 per cent.

RECOMMENDATIONS.

The only recommendations are those given under Study I, page 222. Suggestions as to future work are also made under the several studies.

Mr. **STILWELL.** I would like to suggest that 9 hours' extraction be made official. In ordinary tannery practice it does not seem advisable to extend the time very much over 8 hours, though I think that 10 hours is about the normal practice at present. There is a tendency in some portions of the trade, however, to extend the time of leaching considerably. By continuing the extraction for 24 hours or longer very different results are obtained. As 10 hours is about the general practice, I think approximately that figure could be inserted in the method without any difficulty.

Mr. **VEITCH.** I am heartily in favor of what has been said; in tannery practice 10 hours is sufficient. But the point I want to make, Mr. President, is that it is not wise for us to adopt any change without first trying it.

Mr. **STILWELL.** I agree to that. Ten hours is the normal practice at present. At the same time I am rather anxious to secure some action prohibiting anything over 24 hours' leaching.

Mr. **VEITCH.** This discussion will put the matter on a good basis until some action can be taken. The unnecessary prolonging of the extraction should be stopped.

The **PRESIDENT.** The question is referred to Committee B for consideration as to the advisability of referring the whole matter to the referee for investigation.

The report of Committee A on recommendations, is in order.

REPORT OF COMMITTEE A ON RECOMMENDATIONS (Continued).

B. L. HARTWELL, *Chairman.*

3. NITROGEN.

DETERMINATION OF NITROGEN.

It is recommended—

1. That in the provisional neutral permanganate method the first two lines be changed to read as follows:

> Into a 300 cc low form Griffin beaker weigh 2 grams of the sample, if from a mixed fertilizer; if from concentrated material use a quantity containing approximately 0.075 gram of nitrogen.[a]

2. That the study of methods for the determination of available organic nitrogen be continued.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 67, p. 77, or Bul. No. 73, p. 49.

SEPARATION OF NITROGENOUS BODIES.

Milk and cheese proteids.

It is recommended—

1. That the method given [in the referee's report, page 91] for the separation and determination of casein monolactate and casein dilactate be adopted as a provisional method.

Vegetable proteids.

1. That Mr. Harcourt's report on vegetable proteids be included in the printed proceedings.

Meat proteids.

1. That the following methods be given further trial by the association:

Phosphotungstic acid alone in hot and cold solutions.

Phosphotungstic acid, followed by bromin.

Phosphotungstic acid, followed by zinc sulphate.

Tannin salts, followed by zinc sulphate.

2. That the method for precipitation by bromin alone, and by bromin in the filtrate from zinc sulphate, be discontinued.

4. SOILS.

It is recommended—

1. That in the continuation of the work on methods for available phosphoric acid the referee compare greater concentrations than N/200 acid.

2. That the recommendation calling for a continuation of the work on the solubility of phosphoric acid and potash in the different soil layers and for an investigation of methods for determining total phosphoric acid be left to the discretion of the referee.

3. That the method outlined in the referee's report (p. 145) for the analysis of the water-soluble portion of alkali soils be given further study before its adoption as a provisional method.

4. That on page 72, under 4 (a), acid digestion of the soil, for the words "and again evaporate to complete dryness" substitute the following: Filter, wash free of chlorids, and again evaporate to complete dryness as before.

5. That on page 74, under 4 (g), determination of phosphoric acid, the official method given be marked "(a)" and the following optional provisional method be inserted as (b):

(b) *Optional provisional method:* Proceed as in (a) "until all the phosphoric acid is precipitated," and then finish the determination as follows:

After standing for three hours at a temperature not above 50°, filter on a small filter, wash with water until two fillings of the filter do not greatly diminish the color produced with phenolphthalein by one drop of standard alkali. Place the filter and precipitate in the beaker and dissolve in standard alkali, add a few drops of phenolphthalein solution and titrate with standard acid, 1 cc of which equals 0.0005 gram of phosphoric acid (P_2O_5).

6. That on page 74, 4 (h), the word "available" be omitted from the heading "Provisional method for the determination of available phosphoric acid."

7. That on page 76, paragraph 10, determination of humus, the tenth line, the sentence beginning, "The supernatant liquid is filtered, etc.," shall be changed to read as follows: "The supernatant liquid is filtered, and the filtrate must be perfectly clear and free from turbidity; evaporate an aliquot portion, dry at 100°, and weigh."

8. That the retiring referee be instructed to make such verbal corrections in the methods as these changes will involve, and submit them to the secretary for approval and publication.

RESOLUTION.

W. A. Withers. *Resolved*, That the referee on soils be requested to test, if possible, the value of proposed methods for comparing the ability of different soils to support the growth of nitrifying organisms when the conditions are identical as to temperature, moisture, number of germs, etc.

5. Ash.

It is recommended—

1. That the method for the determination of sulphates, as described on page 25 of the Proceedings of the Nineteenth Annual Convention of this association, 1902,[a] be printed as a provisional method.

2. That the peroxid method of determining sulphates be subjected to further study, with a view to substituting it for the nitric-acid method.

6. Insecticides.

It is recommended—

1. That Method I for total arsenious oxid in Paris green be adopted as an official method, this being a repetition of last year's recommendation.

Adopted as an official method.

2. That the four modifications of the Avery-Beans method for total arsenic in Paris green be tested to determine which is the best modification to be adopted as an optional official method.

3. That the electrolytic method be adopted as the official method for determining copper in Paris green, and in copper carbonate, and that the volumetric methods be further tested, paying particular attention to the remarks of Messrs. Davidson, McBryde, and Allen upon this subject.[b] (Recommended for the first time.)

4. That an attempt be made to so change the procedure in the analysis of London purple that some of the organic matter may be removed. The remarks of Messrs. Davidson and McBryde are of value in this connection.[b]

5. That the volumetric silver-nitrate method be adopted as the official method of determining cyanogen in potassium cyanid and that an N/20 instead of N/10 solution of silver nitrate be used. (Recommended for the first time.)

6. That the methods of examination of soda lye be further tested and compared with gravimetric methods and that the points brought out by Messrs. Davidson and McBryde be given consideration.[b]

7. That the Kissling method for determining nicotin be adopted as an official method. (Recommended for the first time.)

8. That special attention be given to the methods for determining formaldehyde in order to avoid needless repetition of work.

(The report of Committee A was voted upon by sections and as a whole and adopted.)

The President. The report of Committee B is in order.

REPORT OF COMMITTEE B ON RECOMMENDATIONS.

G. E. Patrick, *Chairman.*

(Dairy products, foods and feeding stuffs, sugar, and tannin.)

1. Dairy Products.

It is recommended—

1. That the referee on dairy products be requested to continue the investigation of the effects of preservatives upon the albumin of milk.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 73.

[b] See report on insecticides, page 195.

2. FOODS AND FEEDING STUFFS.

It is recommended—

1. That the referee be requested to study the composition of the ether extract obtained after digesting with pepsin the residue obtained by the official method of fat extraction.

2. That the modified König method for the determination of crude fiber be further investigated, especially to ascertain whether the glycerol-acid mixture hydrolyzes any of the true fiber.

3. SUGAR.

It is recommended—

1. That the associate referee on chemical methods be instructed to cooperate with the International Association for the Unification of Sugar Methods to the end that a uniform method for the determination of reducing sugars be obtained.

2. That an associate referee on methods of analysis of molasses be appointed.

3. That the following methods and changes in methods be adopted:

a. Changes in the official methods given in paragraph 1, page 56, and paragraph 2, page 57, of the Proceedings of this association for 1902.[a]

Adopted as official.

b. The provisional method given in paragraph (f), page 57, of the Proceedings of the association for 1902.

c. Under "Optical methods for the determination of sucrose" omit Clerget's method and Creydt's method (pp. 39 and 40 of Bul. No. 46, Methods of Analysis) and substitute therefor the German official methods for sucrose and raffinose as the official methods of this association. These methods are given on page 57 of the Proceedings of the association for 1902, but in the twenty-fifth line from the top of the page it is recommended to change the words "Dissolve 13.024 grams of the substance in 75 cc of water," so as to read, "Take 50 cc of the clarified solution freed from lead, add 25 cc of water—".

Adopted as official.

d. As an alternate official method for inversion without the application of heat the following is recommended:

Take 50 cc of the clarified solution freed from lead, add 5 cc of hydrochloric acid containing 38.8 per cent of acid, set aside during a period of 24 hours at a temperature not below 20° C.; or if the temperature be above 25° C. set aside for 10 hours. Make up to 100 cc at 20° C. and polarize.

Adopted as official.

e. As provisional methods for the analysis of sugar beets:

Scheibler's alcoholic method and Pellet's aqueous method (p. 58, Proceedings for 1902).

f. As provisional, the methods selected by the international committee for unifying methods of sugar analysis (pp. 58, 59, Proceedings for 1902, pars. 1 to 9, inclusive).

4. That the referees on sugar be requested to continue investigations along the same line as during the past year.

TANNIN.

The following recommendations were not received until after Committee B had finished its work, and they were referred by the association to the referee for 1904:

It is recommended—

1. That the last sentence of Paragraph II, Quantity of Material (Bul. 46, Methods of Analysis, p. 79), be changed to read as follows:

In the case of extract, weigh in a weighing bottle, fitted with a ground-glass stopper, such quantity, etc.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 73.

2. That the last sentence of Paragraph V, Soluble Solids (Bul. 46, Methods of Analysis, p. 80), be changed to read as follows:

> To guard against loss by evaporation, the flask receiving the filtered liquor shall be stoppered with a rubber stopper and the funnel stem passed through the perforation. The funnel shall be covered with a glass plate having an orifice only sufficiently large to admit the air required by the displacement of the liquor.

A motion made by Mr. Stilwell, and amended by Mr. Veitch, to the effect that the time of extraction of spent tan bark be fixed at 10 hours at steam heat, was referred to Committee B, but no action was taken. Aside from the action taken on the tannin recommendations as noted above, the several sections were acted upon and the report adopted as a whole.

The President. The report of Committee C will be received.

REPORT OF COMMITTEE C ON RECOMMENDATIONS.

A. E. Leach, *Chairman.*

(Food adulteration.)

1. Distilled Liquors.

On page 96 of Bulletin No. 65,[a] under Determination of Alcohol, substitute for the first sentence the following: "Weigh or measure (at 15.6° C.) in a distilling flask 20 to 25 cc. of the sample, dilute with 100 cc. of water, and proceed as directed on page 82."

On the same page, under Determination of Extract, strike out the method there given and insert the following: "Weigh or measure (at 15.6° C.) 100 cc of the sample, evaporate nearly to dryness on the water bath, then transfer to a water oven, and dry at the temperature of boiling water."

On the same page, under Determination of Acidity, strike out the method given and insert the following:

> Titrate 100 cc (or 50 cc diluted to 100 cc if the sample is dark in color) with decinormal barium hydrate, using phenolphthalein as indicator. The number of cubic centimeters employed is multiplied by 0.006 for the acidity expressed in grams of acetic acid per 100 cc.

On the same page, under the Determination of Fusel Oil, strike out the third paragraph and substitute the following:

> Add a small quantity of alkali to 200 cc of the sample under consideration and distill slowly, till about 175 cc have passed over, allow the distilling flask to cool, add 25 cc of water, and distill again until the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume[b] (sp. gr. 0.96541 at 15.6°).

On page 98 strike out the method given under Determination of Ethereal Salts and substitute the following:

> Neutralize the residue left after distillation in the fusel oil determination with N/10 H_2SO_4 and add an excess of 10 cc of the acid. Let stand five minutes and make up to 200 cc. Titrate 2 portions of 25 cc each, using as indicators methyl orange in the first and phenolphthalein in the second. The difference gives the amount of alkali necessary to neutralize the organic acids in 25 cc of the sample. By subtracting from this figure the number of cubic centimeters of alkali required for the free acids, and multiplying the result by 0.0088, the number of drams of ethereal salts (calculated as ethyl acetate) in 25 cc of the sample is determined.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Provisional Methods for the Analysis of Foods.

[b] Refers to footnote *b* on p. 96 of Bul. No. 65.

2. DAIRY PRODUCTS.

On page 36, under Detection of Formaldehyde, designate the method there given as (a) *Hehner's method*, and insert the following method, lettered (b):

(b) *The hydrochloric acid and ferric chlorid method.*[a]—To 10 cc of milk in a porcelain casserole add an equal volume of concentrated hydrochloric acid containing 1 cc of 10 per cent ferric chlorid solution to each 500 cc of acid. Heat nearly to the boiling point over the free flame, holding the casserole by the handle and giving it a rotary motion to break up the curd. A violet coloration indicates formaldehyde.

Insert also as (c) the phloroglucin method as described at the foot of page 72.

On page 36, after section 7, "Detection of Borax and Boric Acid," insert the following:

8. DETECTION OF BENZOIC ACID.[b]

Add 5 cc of dilute hydrochloric acid to 50 cc of the milk in a flask and shake to curdle. Then add 150 cc of ether, cork the flask and shake well. Break up the emulsion which forms by aid of a centrifuge, or if the latter is not available extract the curdled milk by gently shaking with successive portions of ether, avoiding the formation of an emulsion.[c] Transfer the ether extract (evaporated to small volume if large in bulk) to a separatory funnel and separate the benzoic acid from the fat by shaking out with dilute ammonia, which takes out the former as ammonium benzoate. Evaporate the ammonia solution in a dish over the water bath till all free ammonia has disappeared, but before getting to dryness add a few drops of ferric chlorid reagent. The characteristic flesh-colored precipitate indicates benzoic acid. Care should be taken not to add the ferric chlorid till all the ammonia has been driven off, otherwise a precipitate of ferric hydrate is formed.

9. DETECTION OF SALICYLIC ACID.

Proceed exactly as directed for benzoic acid. On applying the ferric chlorid to the solution after evaporation of the ammonia, the well-known violet color indicates salicylic acid when present.[d]

On page 36, "Detection of Foreign Colors" should be numbered as "10." On page 39, change heading 8 to "Detection of Foreign Colors" inserting "(a) *Cornwall's method*"[d] before the method as there given.

On page 39, following Cornwall's method, insert the following:

(b) *Method of the Massachusetts Board of Health for annato.*—Treat 2 or 3 grams of the melted and filtered fat (freed from salt and water) with warm, dilute sodium hydroxid, and after stirring pour the mixture while warm upon a wet filter, using to advantage a hot funnel. If annato is present the filter will absorb the color so that when the fat is washed off by a gentle stream of water the paper will be dyed straw color. It is well to pass the warm alkaline filtrate two or three times through the fat on the filter to insure removal of the color. If, after drying the filter, the color turns pink on application of a drop of stannous chlorid solution the presence of annato is assured.

(c) *Geisler's method for azo colors.*[e]—A few drops of the clarified fat are spread out on a porcelain surface and a pinch of fuller's earth added. In the presence of various azo dyes a pink to violet-red coloration will be produced in a few minutes. Some varieties of the fuller's earth react much more readily than others with azo colors.

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[a] An. Rep. Mass. State Board of Health, 1897, p. 558. Food and Drug Reprint, p. 20.

[b] Leach. An. Rep. Mass. State Board of Health, 1902. Food and Drug Reprint, p. 23.

[c] A volume of ether largely in excess over that of the curdled milk has been found to be less apt to form an obstinate emulsion.

[d] These methods for salicylic and benzoic acids while especially applied to milk, from which the ether extracts both fat and preservative, are useful also with modifications for many other food products. The extraction of the ether solution with dilute ammonia, whereby the preservative is removed, permits the subsequent recovery of the ether by distillation.

[e] J. Am. Chem. Soc., 1898, **20**: 110.

(d) *Low's method for azo colors.*[a]—A small amount of the material to be tested is melted in a test tube, an equal volume of a mixture of one part of concentrated sulphuric acid and four parts of glacial acetic acid is added and the tube is heated nearly to the boiling point, the contents being thoroughly mixed by shaking. The tube is then set aside and after the acid solution has settled out it will be found to be colored wine-red in the presence of azo color, while with pure butter fat comparatively no color will be produced.

(e) *Doolittle's method for azo dyes and annato.*[b]—The melted sample is first filtered. Two test tubes are taken and into each are poured about two grams of the filtered fat which is dissolved in ether. Into one test tube 1 or 2 cc of dilute hydrocloric acid are poured, and into the other about the same volume of dilute potassium hydroxid solution. Both tubes are well shaken and allowed to stand. In the presence of azo dye the test tube to which the acid has been added will show a pink to wine-red coloration, while the potash solution in the other tube will show no color. If annato has been used, on the other hand, the potash solution will be colored yellow while no color will be apparent in the acid solution.

Also on page 39 strike out section 9, Detection of Aniline Colors, and in its place insert the following:

9. DETECTION OF BORIC ACID IN BUTTER.

Melt about 25 grams of the sample on the water bath, pour off the fat from the aqueous solution that settles to the bottom of the container, acidify the aqueous solution slightly with hydrochloric acid and test in the usual manner with tumeric paper for boric acid.

On page 40, under Determination of Fat, insert the following heading for the method there given: "(a) *Official method.*[a]"

On page 41, at the end of the first paragraph, insert the following:

(b) *Lythgoe's modification of the Babcock method.*—Weigh accurately about 6 grams of the sample in a tared beaker. Add 10 cc of boiling water and stir with a rod till the cheese softens and an even emulsion is formed, preferably adding a few drops of strong ammonia to aid in the softening and emulsionizing, and keep the beaker in hot water till the emulsion is tolerably complete and free from lumps.

If the sample is a full cream cheese a Babcock cream bottle is employed. The contents of the beaker, after cooling, are transferred to the test bottle as follows: Add to the beaker about half of the 17.6 cc of sulphuric acid regularly used for the test, stir with a rod and pour carefully into the bottle, using the remainder of the acid in two portions for washing out the beaker. Finally proceed as in the Babcock test for milk. Multiply the fat reading by 18 and divide by the weight of the sample taken to obtain the per cent of fat.

On page 41, at the end of the methods for dairy products, insert under the heading "(C) Condensed Milk," the method given on page 28 of the referee's report, 7 sections.

3. SACCHARINE PRODUCTS.

On page 48, under Determination of Commercial Glucose in Molasses Sirups and Honey, make the following changes:

At the end of the second paragraph insert "Express results in terms of commercial glucose polarizing at 175°."

Cancel the third paragraph and substitute the following:

In honey, which is composed largely of invert sugar, much more accurate results are attained by polarizing at a temperature of 87° in a water-jacketed tube an inverted, half normal solution of the sample prepared as follows: Weigh out one-half the normal weight of the sample (13.024 grams) in a 100 cc graduated flask, dissolve in about 70 cc of water, and add 7 cc of concentrated hydrochloric acid; then heat to 68° C. and cool in the usual manner. After inversion add a few drops of phenolphthalein and enough sodium hydroxid to neutralize. Discharge the pink color with a few drops of dilute hydrochloric acid, cool again, add from 5 to 10 cc of alumina cream and make up to the mark. Multiply by 2 the reading at 87° in the 200 mm tube; divide the result by the factor 163 to express the glucose in terms of glucose polarizing at 175°.

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[a] J. Am. Chem. Soc., **20**: 889.

[b] U. S. Dept. Agr., Bureau of Chemistry, Bul. No. 65, p. 152.

Strike out footnote *b*, in the fourth paragraph.

4. Edible Oils and Fats.

On page 26, before heading 5, Determination of Saponification Number and Soluble and Insoluble Acids, insert the Hanus method as a provisional optional method. This method is printed in full in the referee's report, page 63. The principal points in which it differs from the Hübl method are as follows:

Dissolve 13.2 grams of iodin in 1,000 cc glacial acetic acid (showing no reduction with bichromate and H_2SO_4), and add enough bromin to double the halogen content determined by titration. Three cc of bromin is about the proper amount. The iodin may be dissolved by the aid of heat, but the solution should be cold when bromin is added. The solution is used in the same manner as in the Hübl method except that an excess of 70 per cent of the iodin is allowed; 10 cc of the KI solution is added instead of 20 cc and the time of reaction is shortened to thirty minutes. When the KI solution is added, shake the mixture thoroughly before adding the water.

On page 32, under heading 16, Halphen reaction for cotton-seed oil, third line, insert before the word "brine" the word "saturated," and for "fifteen minutes" substitute "an hour to 2 hours."

On page 33, under heading 18, Renard's Test for Peanut Oil, etc., substitute the method printed in full in the referee's report on page 64. This method is essentially the same as that given on page 33, except that 20 grams of fat are taken for analysis with the proportional increase of reagents rendered necessary by the larger amount of fat. An additional washing is also provided for in the revised method.

In Bulletin 46, page 49, under Determination of Refractive Index, second paragraph, line 3, change the factor 0.000176 to 0.000365, in accordance with the more correct factor given in Bulletin 65. Also cancel the example following and substitute the example given on page 22 of Bulletin 65, under Determination of Index of Refraction. (Recommended for the first time.)

5. Spices.

On page 58 of Bulletin 65, under Determination of Starch by the Diastase Method, fifteenth line, strike out the words "copper reduced by" and insert therefor "dextrose resulting from the inversion of."

6. Fruits and Fruit Products.

The following changes in the provisional methods as given in Bulletin 65 are recommended:

(1) On page 78, under heading 12, Polarization, substitute the method given on page 57 of the Proceedings for 1902, under Determination of sucrose in absence of raffinose.

(2) On page 78, section 13, b, line 8, change "1 per cent" to "0.50 per cent."

(3) On page 78, section 14, lines 8 and 9, for "use Allihn's method for the determination (p. 49) [a]," substitute "Use the official method for the determination of invert sugar as given in Bul. 46, p. 33 (c) (1) (a) and (b)."

(4) On page 78, strike out section 15, Determination of Dextrin, and substitute therefor Determination of Glucose, referring to method given on page 48, section 12.

7. VINEGAR.

On page 65 of Bulletin 65, following section 13, Detection of Coloring Matters, insert Crampton and Simon's method for the detection of caramel as follows: [a]

Add 25 grams of fuller's earth to 50 cc of the vinegar under examination, beat the mixture up in a beaker and let it stand covered half an hour at room temperature, then filter. The determination of the figure representing the color is made with the tintometer upon the liquid before and after treatment and the difference between the two results gives the percentage of color absorbed.

8. COCOA AND COCOA PRODUCTS.

The report submitted is to be printed separately and distributed to the associate referees on food adulteration for criticism before it is printed in the Proceedings, in accordance with the plan previously pursued.

(The recommendations of Committee C were adopted by the association.)

The **PRESIDENT.** If there is no other business before the association, I will declare the convention adjourned sine die.

ERVIN E. EWELL, until recently assistant chief of the Bureau of Chemistry of the Department of Agriculture, and since 1889 a member of that Bureau and of the association, died at New Orleans, La., February 7, 1904, after a brief illness of typhoid fever.

During his fourteen years' service in the Bureau Mr. Ewell accomplished an immense amount of routine work in addition to the research work for which he was especially fitted. These researches included a chemical and physiological study of the mescal button, used by the Mexican Indians, which produces intoxication of a peculiar character; a series of experiments on the nitrifying ferments of soils; miscellaneous studies of a varied character, and investigations of the quality and character of supplies furnished the different branches of the Government, which work has resulted in the establishment of a contracts laboratory in the Bureau of Chemistry. In this work Mr. Ewell's ability was most marked, requiring as it did not only a high degree of chemical skill but also the development of special methods of investigation and great ingenuity in the application of tests.

Mr. Ewell served the association twice as referee on sugar, was vice-president of the Washington section of the American Chemical Society, and had published a number of papers in the journal of that body and in official bulletins of the Department of Agriculture.

In March, 1903, Mr. Ewell resigned from the Bureau of Chemistry to enter a wider field of activity offered by the German Kali Works, with headquarters at Atlanta, Ga., taking with him the best wishes of his associates in the Department. Indefatigable industry, combined with a thorough educational equipment and a fine mentality, had marked Mr. Ewell among the rising young chemists of the day, and science has lost a most promising devotee in his death.

NORMAN ROBINSON, formerly an active member of this association, died in Orlando, Fla., on February 4, 1904. Professor Robinson had served the association as referee on potash in 1903, and to his services as a chemist added those of an educator, being an ardent classical scholar as well as a lover of science. Though in astronomy, botany, and geology he was well versed, it was to chemistry that he gave the most attention, working in his private laboratory in Jacksonville in addition to his professional duties. The association has lost in his death a most able and scholarly member of the profession, and to his family all those who remember Professor Robinson will extend their sympathy in the loss of his companionship. High principles, a mind at once keen and broadly cultured, and a charming personality made Professor Robinson a man to be long and lovingly remembered by those who knew him even slightly.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1904.

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  Separation of nitrogenous bodies:
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    Vegetable proteids: J. S. Chamberlain, Washington, D. C.
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- (2) Saccharine products, including confectionery: E. B. Kenrick, Winnipeg, Manitoba, Canada.
- (3) Fruit products: E. M. Chace, Washington, D. C.
- (4) Wine: G. E. Colby, Berkeley, Cal.
- (5) Beer: H. E. Barnard, Concord, N. H.
- (6) Distilled liquors: C. A. Crampton, Washington, D. C.
- (7) Vinegar: Richard Fischer, Madison, Wis.
- (8) Flavoring extracts: R. E. Doolittle, Lansing, Mich.
- (9) Spices: A. L. Winton, New Haven, Conn.
- (10) Baking powder and baking chemicals: R. O. Brooks, Trenton, N. J.
- (11) Meat and fish: M. E. Jaffa, Berkeley, Cal.
- (12) Fats and oils: L. M. Tolman, Washington, D. C.
- (13) Dairy products: A. E. Leach, Boston, Mass.
- (14) Cereal products: A. McGill, Ottawa, Canada.
- (15) Infants and invalids' foods: H. W. Wiley, Washington, D. C.
- (16) Vegetables: F. W. Bedford, St. Paul, Minn.
- (17) Condiments other than spices: J. D. Hird, Washington, D. C.
- (18) Cocoa and cocoa products: E. N. Eaton, Chicago, Ill.
- (19) Tea and coffee: H. C. Lythgoe, Boston, Mass.
- (20) Preservatives: W. G. Bigelow, Washington, D. C.

Sugar:

Molasses methods: H. E. Sawyer, Boston, Mass.

Special analytical methods: C. A. Browne, jr., Audubon Park, La.

Tannin: H. C. Reed, Stamford, Conn.

Insecticides: S. Avery, Lincoln, Nebr.

Ash: F. T. Shutt, Ottawa, Canada.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statement of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. All persons eligible to membership shall become members ex officiis and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the executive committee, at the regular annual meeting, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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U. S. DEPARTMENT OF AGRICULTURE,

BUREAU OF CHEMISTRY—BULLETIN NO. 82.

H. W. WILEY, CHIEF OF BUREAU.

PARIS GREEN SPRAYING EXPERIMENTS.

BY

J. K. HAYWOOD,

Chief Insecticide and Agricultural Water Laboratory,

**In collaboration with the Division of Entomology, U. S. Department of Agriculture, and
the agricultural experiment stations of Maryland, New York,
New Hampshire, Rhode Island, South
Carolina, and Oregon.**

WASHINGTON:
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LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., March 19, 1904.

Sir: I have the honor to transmit for your approval a report on a series of spraying experiments with Paris Green, prepared in the insecticide and agricultural water laboratory of this Bureau, and to recommend its publication as Bulletin No. 82 of the Bureau of Chemistry. It is desired to acknowledge especially the cooperation of the Division of Entomology and the contributions of the various experiment stations named, at which so large a part of the work was performed. The two half-tone plates express graphically one of the most important points made in the bulletin and are deemed of value in impressing the conclusions drawn in the text.

Respectfully,

H. W. Wiley, *Chief.*

Hon. James Wilson,
Secretary of Agriculture.

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PARIS GREEN SPRAYING EXPERIMENTS.

REASONS FOR THE INVESTIGATION.

These experiments were undertaken to show how much soluble arsenious oxid may be present in samples of Paris Green used for spraying purposes without injury to the foliage. As is well known, Paris Green is composed of copper oxid, acetic acid, and arsenious oxid, combined as copper-aceto-arsenite in the following proportions:

	Per cent.
Copper oxid	31.29
Arsenious oxid	58.65
Acetic acid	10.06

Since this substance is always sold upon the market as a commercial rather than a chemically pure article, small amounts of impurities are always present. Among these may be mentioned sand, sodium sulphate, and arsenious oxid, the last of which is not combined as it should be with the other constituents, but is present in the free state. It is to this impurity of Paris Green that the scorching properties are usually ascribed. California has passed a law limiting the amount of free arsenious oxid in Paris Green to 4 per cent, while New York's law limits the amount to 3.5 per cent. In the other States, where there are no laws governing the matter, the entomologists usually consider 4 per cent of free arsenious oxid to be the maximum amount allowable in Paris Green used for spraying.

It does not seem to have been generally recognized by entomologists[a] and chemists that there may be three conditions of Paris Green which will cause a scorching of the foliage.

(1) As indicated above, there may be a certain amount of arsenious oxid in Paris Green over and above that combined with the other con-

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[a] Marlatt in two articles (Insect Life, vol. vii, July, 1895, pp. 408–411, and bulletin No. 6, Division of Entomology, September, 1896, pp. 30–35) has recognized the first and third conditions of Paris Green that may give rise to a scorching of the foliage, but not the second condition.

stituents. This is "free" arsenious oxid and until recently it has been considered the only cause of a scorching of the foliage by Paris Green.

(2) The greens may be poorly made so that the constituents are very loosely held together. When such greens are brought in contact with water, especially water containing carbon dioxid, they are soon broken up and arsenious oxid is set free. Even the best greens break up to some extent under such conditions, but the poorly made greens decompose much more rapidly. Note, then, the effect of such greens upon the foliage. First, the water of the spray would act at once on the green and set arsenious oxid free, then carbon dioxid would dissolve in the water and carry the decomposition of the green still further. But the matter would not end here, for the rain and dew both containing carbon dioxid would also act upon the green and enough free arsenious oxid would soon collect to severely scorch the foliage. This would of course take place more slowly with even the best greens, but unless the climatic conditions were very adverse, not enough free arsenious oxid would be apt to accumulate at one time to cause any serious damage.

(3) The green may be extremely fine. It is well known that even the best greens when ground to a very fine powder and applied to the foliage will scorch. This is doubtless due to the fact that more surface is exposed to the action of water, which may contain carbon dioxid, so that its decomposing action on the green is accelerated and enough free arsenious oxid soon gathers to cause serious damage. Avery and Beans[a] found that the very best Paris Greens obtainable were slowly attacked by water, and by grinding the same greens to a very fine powder and suspending them in water the rate of decomposition was enormously increased. They also found that carbon dioxid in the water increases the rapidity of decomposition.

It is a very common occurrence to secure a commercial Paris Green that scalds because of one of the first two causes—i. e., either by reason of the presence of free arsenious oxid or because it is poorly manufactured—but the writer has never found a commercial sample of green that scorched because it was in too fine a condition. In experimenting on the third method of scorching even the finest samples of green found on the market had to be ground further before they would scorch by reason of minute division.

It is thus seen that the chemist will be called upon to examine samples of green (1) for actual free arsenious oxid; (2) for arsenious oxid that may be easily set free on account of the instability of the compound. Methods of determining these two forms of soluble arsenious oxid have been devised during the last few years which, though not strictly accurate, will give results close enough for all practical purposes. These methods, with the reason for their adoption, are as follows:

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[a] J. Amer. Chem. Soc., 23: 111.

In determining soluble arsenic it is evident that it would not be sufficient to extract the green for only 24 hours with water, since in samples containing free arsenious acid as such, all of the arsenic does not go into solution in that length of time, and neither do samples that are poorly made break up to any extent within twenty-four hours. The author has found that it takes from five to ten days, according to the amount present, for all the free arsenious oxid to go into solution. It has also been found that badly made greens are broken up enough during the course of ten days to give some idea of their instability and that well-made greens are not broken up to any great extent during that length of time. It has therefore been thought best to determine the soluble arsenious oxid in Paris Greens by extracting one part of the green with 1,000 parts of carbon dioxid-free water for ten days and at the end of this time determining the arsenious oxid in an aliquot portion of the filtrate. This method shows whether the green contains a large quantity of free arsenious oxid or whether it is badly made, but does not distinguish between the two. A method devised by Avery and Beans,[a] however, allows us to approximately determine the amount of free arsenious oxid present exclusive of that formed by the decomposition of the green. Therefore, by using both methods the chemist will be able to tell either the amount of free arsenious oxid actually present in the green or the amount that is apt to be present a short time after spraying. Both of these forms of soluble arsenious oxid are undoubtedly injurious, although perhaps not equally so.

These two distinct types of soluble arsenious oxid in commercial Paris Green have not been previously recognized, and furthermore when 4 per cent was adopted as the maximum amount of soluble arsenious oxid allowable in Paris Green the extraction with water was continued for a much shorter time, resulting in much lower figures for soluble arsenious oxid. It would appear, therefore, that investigation along this line is needed[b] to determine the maximum amount of soluble arsenious oxid, as found by our present methods of analysis, that can be present in a Paris Green in both forms without danger of scorching. This investigation is an attempt to answer the question under the first condition, i. e., when the soluble arsenious oxid is present in the free condition. It is hoped that in the near future experiments may be made on Paris Greens of the second type.

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[a] J. Amer. Chem. Soc., 23: 111.

[b] Valuable work upon the effect of arsenic and arsenite on plants has been published by Gillette, Woodworth, Bailey, Marlatt, and others, but not along the lines followed in this experiment.

PRELIMINARY WORK AT WASHINGTON, D. C.

PLAN OF THE EXPERIMENT.

Preliminary work was begun during the early summer of 1902, and carried out in the following manner: A large quantity of Paris Green, as pure as could be obtained, was purchased and a determination made of the soluble arsenious oxid by the ten-day water-extraction method. Then known quantities of perfectly pure arsenious oxid were added to different quantities of the green, so that samples of a definite arsenious oxid content were obtained. Each of these samples contained to 1 pound of Paris Green the following amounts of soluble arsenious oxid:

	Per cent.
No. 1	4.29
No. 2	5.00
No. 3	6.00
No. 4	7.00
No. 5	8.00

Two sets of these samples were made up and applied in the following manner:

SET I.

Treat one-half of each of 5 samples described above with 150 gallons of water, making 1 pound to 300 gallons, and spray delicate trees, such as peach trees.

Treat the other half of each of the 5 samples with 150 gallons of water, making 1 pound to 300 gallons, and 1 pound of lime, and again spray delicate trees, such as peach trees.

SET II.

Treat one-half of each of above 5 samples with 80 gallons of water, making 1 pound to 160 gallons, and spray hardy trees, such as apple trees.

Treat the other half of the 5 samples with 80 gallons of water, making 1 pound to 160 gallons, and 1 pound of lime, and again spray hardy trees, such as apple trees.

The dates of spraying and taking of observations and the duration of the experiment were carefully noted.

With the aid of Mr. F. C. Pratt, an assistant in the Division of Entomology, the author sprayed young peach, apple, and pear trees, following very closely the directions outlined above. The trees grew in the Agricultural Department grounds, and, unfortunately, had been used before in various experiments upon insects and sprays, so that all of them were not in a perfectly healthy condition. Since they were the only trees available, and the investigation was only preliminary in character, to fix within wide limits the amount of soluble arsenic that can be endured by the trees, it was thought they would serve the purpose. The table on the next page, giving the weather conditions during the first part of the experiment, is furnished by the United States Weather Bureau.

Weather conditions, Washington, D. C., June 20 to July 15, 1902.

Date.	Mean temperature.	Precipitation.	General character of day.
1902.	° *F.*	*Inches.*	
June 20	78	Trace.	Partly cloudy.
21	73	0.85	Cloudy.
22	64	.00	Clear.
23	64	Trace.	Partly cloudy.
24	60	.00	Do.
25	64	.01	Clear.
26	73	1.37	Do.
27	72	.00	Partly cloudy.
28	70	.01	Cloudy.
29	80	.03	Partly cloudy.
30	66	.31	Cloudy.
July 1	68	[a].01	Clear.
2	72	.00	Do.
3	82	.06	Partly cloudy.
4	80	Trace.	Do.
5	84	.00	Do.
6	84	Trace.	Do.
7	74	Trace.	Cloudy.
8	81	.00	Partly cloudy.
9	82	.00	Do.
10	78	.34	Do.
11	70	.00	Clear.
12	70	.00	Do.
13	72	.00	Do.
14	79	.00	Do.
15	82	.02	Do.

[a] In the morning, before the trees were sprayed.

OBSERVATIONS.

The trees were first sprayed on June 20. The next day a heavy rain fell, which seemed to wash off the Paris Green completely. After waiting for 10 days—i. e., until July 1—to see whether the trees would be affected, they were again sprayed, no damage being observed at that time. After the second spraying observations were made on the following dates: July 5, 10, 16, August 11, and September 18, 1902.

With the peaches the results were somewhat irregular on account of the varying hardiness of the trees and the fact that some of them were attacked later in the season by insect pests; but a few general conclusions can be drawn. As one would expect from previous experiments, the peaches did not show any injury from the spray until after the first considerable rain, on July 10. Then the trees on which lime was not used were all more or less burned and partly defoliated, except the one sprayed with solution No. 3 (6 per cent of arsenious oxid). This tree was very hardy and was not injured in any way. The tree sprayed with solution No. 1 (4.29 per cent of arsenious oxid), without lime, was not injured to a great extent, but a few leaves fell. The injury to trees when lime was used with the Paris Green was not nearly so great as when lime was not used, which was to be expected. Those sprayed with solutions Nos. 1, 2, and 3 (4.29, 5, and 6 per cent of arsenious oxid), with lime, while showing a slight yellowing, spotting, and falling of the leaves, were not injured to any great extent, and the falling of the leaves soon ceased. All of the sprayed peach trees lost their leaves much earlier in the autumn than is common in and around Washington; but as all of the trees near by had been used in the experiment it was not possible to make any comparison with unsprayed trees to ascertain whether the premature ripening of the leaves was due to some other cause than the poison.

The apple trees when sprayed with varying strengths of arsenious oxid, without lime, all showed about 1 to 2 per cent of injury. A few leaves were curled and burned at the edges where the Paris Green had collected in the greatest quantities, but the injury was slight, and after these leaves fell the trees did not show that they had been sprayed at all. All were in good condition and remained so throughout the summer, except the one sprayed with solution No. 1, which died during August.

The same results were obtained on the trees sprayed with the various solutions after the addition of lime. None was seriously injured, and in about two weeks after the spraying no effects could be seen. In the case of the tree sprayed with solution No. 2, with lime, it was observed that about one-third of the leaves had fallen on September 18, but as the trees sprayed with much stronger solutions were in no way affected it appeared that the dropping of the leaves was not caused by the spray. It was noted in the case of trees sprayed both with and without lime that about one-half of them lost the greater part of their leaves by October 12, which is rather early in this climate. This was not true to so great an extent of the trees that had been sprayed with the addition of lime, but still there was a tendency in this direction. Four extremely hardy apple trees, sprayed with solutions Nos. 3 and 4 (6 and 7 per cent arsenious oxid), with lime, did not drop their leaves early, while two trees, not so hardy, that were sprayed with solutions Nos. 1 and 2 (4.29 and 5 per cent arsenious oxid) dropped over one-half of their leaves by October 12. It did not appear that the premature dropping of the leaves in the autumn was accompanied by any permanent injury to the tree.

The pear trees, in those cases in which no lime was used, had a few leaves burned on the edges at the first and second observations, but these soon dropped off or withered away, and the trees were in excellent condition. In fact, it may be said that pear trees sprayed with any of the five solutions without lime were practically unaffected, none being damaged to the extent of 2 per cent. The trees sprayed with any one of the solutions with lime were not scorched in the slightest degree.

Toward the end of the season the tree treated with solution No. 1 (4.29 per cent of arsenious oxid), without lime, died, and the tree treated with solution No. 2 (5 per cent of arsenious oxid), with lime, dropped its foliage prematurely, but this was not attributed to the spray, since trees treated with much stronger solutions were in excellent condition, and, furthermore, the injured trees appeared to have been attacked by pear-leaf blight, so that a large number of leaves turned black and withered.

SUMMARY OF RESULTS.

Under the conditions prevailing during the early summer of 1902 at Washington, D. C., the following conclusions may be drawn:

(1) If lime is not used peach trees can not be sprayed with Paris Green containing even as little as 4.29 per cent of soluble arsenious oxid; if lime is used greens containing as much as 6 per cent of soluble arsenious oxid may be used without causing serious damage.

(2) Apple trees may be sprayed with Paris Green containing as much as 8 per cent of soluble arsenious oxid, either with or without lime, without any immediate injury to the tree, although what the effect might be in causing the trees to drop their leaves prematurely and expose the fruit has not been determined.

(3) Pear trees may be sprayed with Paris Green containing as much as 8 per cent of soluble arsenious oxid, either with or without lime, without any injury to the tree.

ORGANIZATION OF COOPERATIVE WORK.

During the spring of 1903 the author addressed a letter to the entomologists of several of the experiment stations in different parts of the country asking for their cooperation in a continuation of this work. Favorable replies were obtained from about ten different stations, but reports were received only from the following: John Craig and H. E. Weed, Ithaca, N. Y.; C. M. Weed and J. L. Randall, Durham, N. H.; F. W. Card and A. E. Stene, Kingston, R. I.; C. E. Chambliss, Clemson College, S. C.; and A. B. Cordley, Corvallis, Oreg. Since the fruit trees could not be obtained in Washington for spraying, permission was obtained of Director Patterson, of the Maryland Agricultural Experiment Station at College Park, to use trees upon the experimental farm. The spraying and taking of notes was done by Mr. Smith, of the Bureau of Chemistry, and Mr. Symons, of the Maryland station.

PREPARATION OF SAMPLES.

As in the previous year's work, a large keg of as fine Paris Green as it was possible to obtain was purchased and subjected to a chemical examination. It was found that 2.84 per cent of soluble arsenious oxid was present, according to the ten-day water-extraction method. Definite quantities of chemically pure arsenious oxid were added and four sets of 6 samples each of Paris Greens were obtained and sent to all the collaborators. Each sample weighed 1 ounce and contained the following amounts of soluble arsenious oxid:

	Per cent.
No. 1	2.84
No. 2	4.00
No. 3	5.00
No. 4	6.00
No. 5	7.00
No. 6	8.00

DIRECTIONS FOR SPRAYING.

(1) Treat each of the 6 samples with 9.5 gallons of water (approximately 1 pound to 150 gallons) and spray hardy trees such as apples and pears.

(2) Treat each of the 6 samples with 9.5 gallons of water and 2 ounces of previously slaked lime, and again spray hardy trees such as apples and pears.

(3) Treat each of the 6 samples with 15.5 gallons of water (approximately 1 pound to 250 gallons) and spray delicate trees such as peaches and plums.

(4) Treat each of the 6 samples with 15.5 gallons of water and 2 ounces of previously slaked lime, and again spray delicate trees such as peaches and plums.

Make duplicate and triplicate experiments in each case if enough trees are available. When possible tell what varieties of trees were sprayed.

Carefully note the date of spraying and observe the trees for at least three weeks. Also note whether the leaves fall from these trees earlier in the fall than from similar trees that were not sprayed. Keep a record of the weather conditions during the experiment.

Always use the whole of the 1-ounce sample forwarded to you with the required amount of water. This precaution is of the greatest importance, because of the fact that these 1-ounce samples are prepared from Paris Green to which is added definite amounts of pure arsenious oxid in order to obtain the various percentages of this substance. These are mixed as thoroughly as possible, but such mixing can never be perfect. Therefore, if only a part of the sample were used a higher or lower percentage of arsenious oxid might be present in that part than in the whole sample.

REPORT FROM THE MARYLAND STATION, COLLEGE PARK.

By B. H. Smith, T. B. Symons, and J. K. Haywood.

Forty-eight trees were utilized for this experiment, two for each of the solutions tested, one with and one without the addition of lime. The apple trees were York Imperials; the pears, Keiffers; the plums, American, and the peaches, Elberta and Mountain Rose. They were all in good condition and from 5 to 10 years old.

The following table gives a description of the weather conditions during the experiment:

Meteorological conditions at College Park, Md.

Date.	Precipitation.	General character of the day.	Date.	Precipitation.	General character of the day.
1902.	*Inches.*		1902.	*Inches.*	
May 11	0.00	Partly cloudy.	May 26	0.00	Partly cloudy.
12	.00	Clear.	27	1.53	Do.
13	.00	Do.	28	Trace.	Do.
14	.00	Do.	29	.00	Do.
15	.00	Do.	30	.81	Do.
16	.00	Do.	31		
17	.00	Do.	June 1	.00	Do.
18	.00	Do.	2	.00	Clear.
19	.00	Do.	3	.00	Do.
20	.00	Do.	4	.00	Do.
21	.00	Do.	5	.00	Partly cloudy.
22	.00	Partly cloudy.	6	1.07	Do.
23	.36	Do.	7		
24			8	.00	Do.
25	.00	Do.	9	.00	Do.

OBSERVATIONS.

The spraying was done on May 11, 1903, and the first observation was made on May 18, no rain having fallen in the meantime. No injurious effect from the sprays could be discovered on any of the trees. The orchard was examined a second time on May 26, a light rain having fallen on May 24. Among the apple trees a small percentage of the leaves of Nos. 4, 5, and 6 (6, 7, and 8 per cent of arsenious oxid, without lime) were very slightly burned, while No. 5 (7 per cent of arsenious oxid, with lime) had an occasional blackened leaf. The remainder showed no injury.

A number of the pear trees were affected with leaf spot (*Septoria pyricola*) and the pear leaf blister mite (*Phytoptus pyricola*) so that a great many leaves were beginning to blacken and wither.

A small part of the foliage of all the peach trees used in the experiment had begun to turn yellow and an occasional leaf showed black spots, but the general effect at this time was so slight that no appreciable difference could be said to exist between the several trees.

The plum trees showed no injurious effect from the spraying, with the exception of No. 5 (7 per cent of arsenious oxid, without lime), on which a few leaves were spotted.

On June 9, after three more rains, the apple trees had undergone practically no change since the first observation.

On the pears the disease and mite before mentioned were working havoc, and as the injury was as great on the trees sprayed with the weaker as with the stronger solutions and several trees not sprayed at all were affected to nearly as great an extent as those sprayed, it was thought best to exclude the pear trees from the experiment.

The peaches were now considerably damaged, though how much of this was due to the rather dry weather of the previous month, which doubtless had a certain amount of influence, was difficult to determine. The notes taken at this time are given below:

PEACHES (WITHOUT LIME).

No. 1. A few leaves have black spots; about 35 per cent turning yellow and dropping.

No. 2. Same.

No. 3. About 5 per cent of leaves more or less scorched; half the leaves dropping.

No. 4. Same.

No. 5. About 10 per cent of the leaves are scorched; leaves are yellow and as many on the ground as on the trees.

No. 6. Not burned as badly as the others and fewer leaves dropping; a very vigorous tree.

PEACHES (WITH LIME).

No. 1. No leaves burned; about 5 per cent dropping.

No. 2. Same, except that about 10 per cent are dropping.

No. 3. Very few leaves burned; leaves dropping considerably.

No. 4. Injury a little more pronounced than on No. 3.

No. 5. Practically the same as No. 4.

No. 6. About 5 per cent of leaves burned and dropping; almost the same as Nos. 4 and 5.

At this time the plum leaves were perforated to some extent by the plum leaf shot-hole fungus (*Septoria cerasina*), but also showed considerable injury from the spray. The notes made on this date are as follows:

PLUMS (WITHOUT LIME).

No. 1. Very few leaves burned.
2. Probably 5 per cent of leaves burned.
3. Same as No. 1.
4. Large percentage of leaves burned at edges.
5. Large percentage of leaves burned at edges.
6. Large percentage of leaves burned at edges.

PLUMS (WITH LIME).

No. 1. Few leaves slightly burned; small tree.
2. No injury.
3. No injury.
4. Few leaves burned.
5. Few leaves burned.
6. Few leaves burned.

After twenty days another examination was made. The intervening wet weather was evidently of great benefit to the orchard, as the trees had begun a new growth and the leaves developed a darker color.

The slight injury to the apple trees of the month before was not noticeable, both those sprayed with and without lime being in good condition.

The peaches were located on a dry knoll and accordingly had suffered most from the drought, but at this observation, after having dropped their affected leaves and put forth new ones, both series of the trees had so far recuperated as to show scarcely any effect from the Paris Green, the foliage being nearly as thick as upon unsprayed trees.

The plum trees, while showing more injury than the peaches, were decidedly improved.

The last examination was made on August 8, and as far as could be ascertained the trees of all varieties had completely recovered from the effects of the various sprays.

CONCLUSIONS.

The effect of these Paris Greens, both with and without lime, upon the apple trees was very slight, and, as far as could be estimated, the same may be said of the pears.

To the peach trees considerable temporary injury, as shown by a dropping of the leaves, was done by all the greens without lime, although the trees seemed to recover later in the season. With lime the peaches were not injured to any great extent, even temporarily, by greens Nos. 1, 2, and 3 (2.84, 4, and 5 per cent of arsenious oxid), but were injured temporarily to quite an extent by greens Nos. 4, 5, and 6 (6, 7, and 8 per cent of arsenious oxid). All of the trees, however, seemed to recover before the end of the season. An interesting point noted in the case of the peach trees was that no injury was noticeable until a month after the application of the spray, the delay in action being probably due to the absence of rain.

Another fact emphasized by the observations was that the injury always depended to a considerable extent upon the physical condition of the individual tree; in a number of cases a particularly vigorous specimen showing no injury, while its less thrifty neighbor, which was perhaps sprayed with a Paris Green containing less soluble arsenious oxid, was seriously damaged.

From the results of this work it is believed that apple and probably pear trees can be sprayed with Paris Green containing 8 per cent of soluble arsenious oxid and sustain no injury. The observations made on peach and plum trees tend to show that if lime is added Paris Green containing as high as 5 per cent of free arsenious oxid may be safely used.

REPORT FROM THE NEW YORK STATION, ITHACA.

By JOHN CRAIG and H. E. WEED.

The Paris Green was applied to the foliage of apple, pear, peach, and plum trees according to the directions accompanying the samples. The trees were sprayed on May 25, and 26, 1903, and the final results observed and notes taken on June 16. In the notes on the damage done to the foliage given below the terms "no injury indicated," "injury indicated," "somewhat injured," "injured," and "badly injured" are used to indicate the various degrees of injury in order that the results may be compared. In the same way the terms "spraying safe," "spraying questionable," and "spraying unsafe" are employed to indicate whether or not it would be expedient to spray with the amount of soluble arsenious oxid contained in the solution.

The rainfall during the period of the experiment and the results obtained are as follows:

Rainfall at Ithaca, N. Y.

Date.	Precipitation.	Date.	Precipitation.	Date.	Precipitation.	Date.	Precipitation.
1903.	*Inches.*	1903.	*Inches.*	1903.	*Inches.*	1903.	*Inches.*
May 1	T.	May 15	.00	May 29	.01	June 12	1.66
2	0	16	.00	30	.00	13	.01
3	.03	17	.00	31	.00	14	.01
4	.13	18	.00	June 1	.00	15	.11
5	0	19	T.	2	.00	16	T.
6	0	20	.00	3	.00	17	.19
7	T.	21	.02	4	.00	18	.28
8	0	22	T.	5	.00	19	.09
9	0	23	.00	6	.00	20	1.06
10	0	24	T.	7	.03	21	.74
11	0	25	.00	8	.01	22	0
12	0	26	T.	9	.05	23	.54
13	0	27	T.	10	.02		
14	0	28	.06	11	.04		

OBSERVATIONS.

APPLE, VARIETY GOLDEN SWEET.

No.	Mixture used.	Effect on foliage.	Remarks.
1	Without lime	Injury indicated	Spraying safe.
	With lime	No injury indicated	Do.
2	Without lime	Injury indicated	Do.
	With lime	No injury indicated	Do.
3	Without lime	Somewhat injured	Spraying questionable
	With lime	Injury indicated	Spraying safe.
4	Without lime	Injured	Spraying unsafe.
	With lime	Slight injury	Spraying safe.
5	Without lime	Injured	Spraying unsafe.
	With lime	Somewhat injured	Spraying questionable.
6	Without lime	Badly injured	Spraying unsafe.
	With lime	Injured	Do.

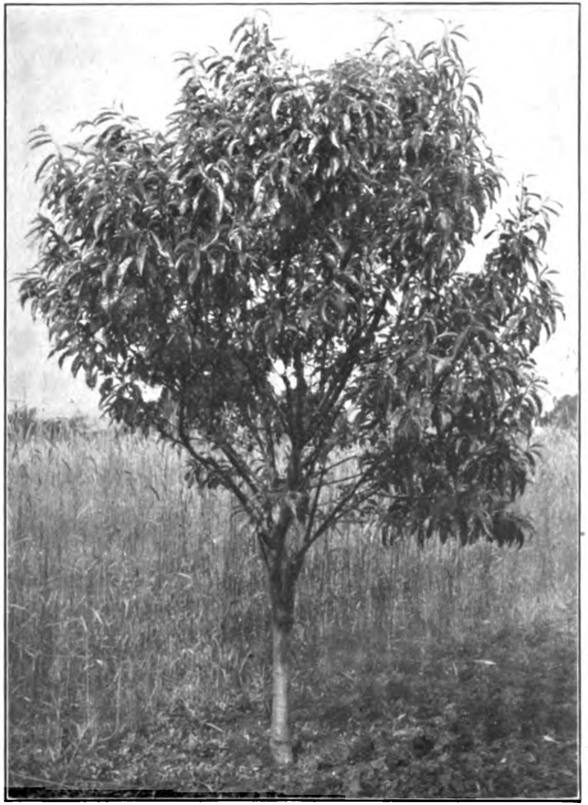

FIG. 1.—PEACH TREE WITH FOLIAGE IN NORMAL CONDITION.

Kingston, R. I., June 18, 1903.

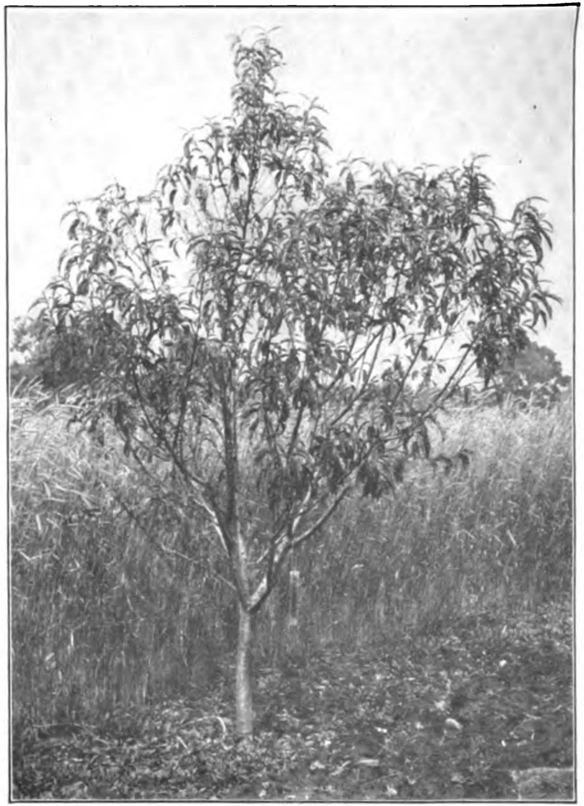

FIG. 2.—PEACH TREE SPRAYED WITH SOLUTION NO. 1, WITHOUT LIME.

Kingston, R. I., June 18, 1903.

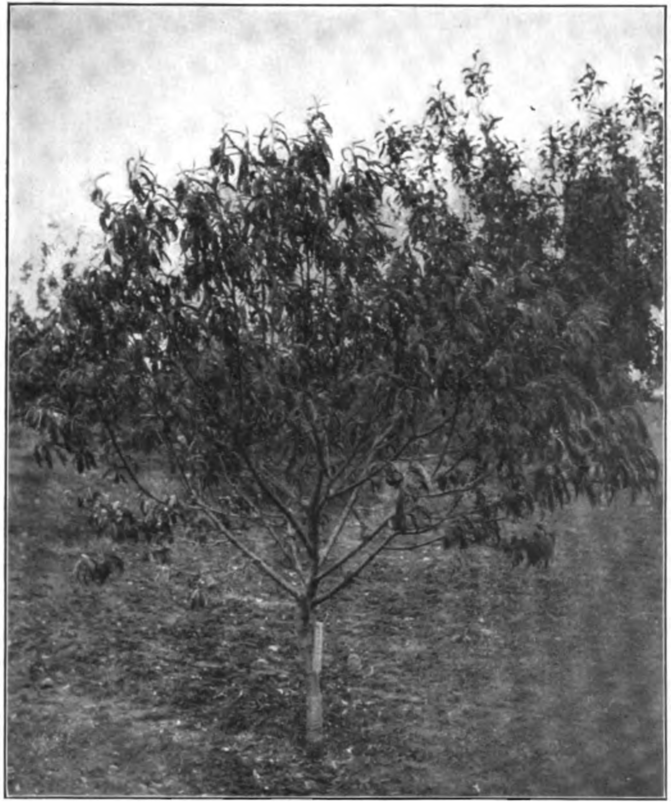

FIG. 1.—PEACH TREE SPRAYED WITH SOLUTION NO. 6, WITH LIME.

Kingston, R. I., June 18, 1903.

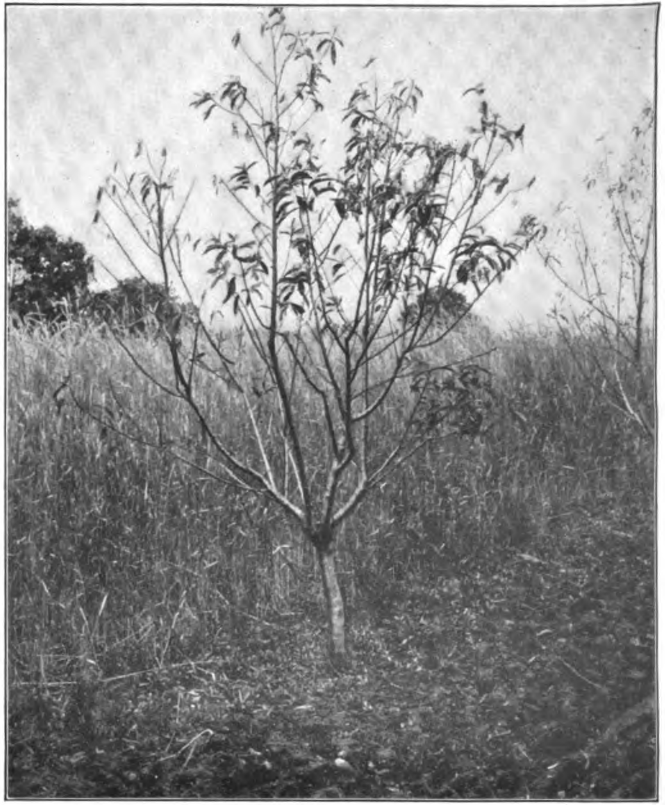

FIG. 2.—PEACH TREE SPRAYED WITH SOLUTION NO. 6, WITHOUT LIME.

Kingston, R. I., June 12, 1903.

Observations—Continued.

PEAR, VARIETY BARTLETT.

No.	Mixture used.	Effect on foliage.	Remarks.
1	Without lime	Injury indicated	Spraying safe.
	With lime	No injury indicated	Do.
2	Without lime	Somewhat injured	Spraying questionable.
	With lime	Injury indicated	Spraying safe.
3	Without lime	Injured	Spraying unsafe.
	With lime	Injury indicated	Spraying safe.
4	Without lime	Injured	Spraying unsafe.
	With lime	Somewhat injured	Spraying questionable.
5	Without lime	Badly injured	Spraying unsafe.
	With lime	Injured	Do.
6	Without lime	Badly injured	Do.
	With lime	Injured	Do.

PLUM, VARIETY MARIANNA.

No.	Mixture used.	Effect on foliage.	Remarks.
1	Without lime	Injury indicated	Spraying safe.
	With lime	No injury indicated	Do.
2	Without lime	Injured	Spraying questionable.
	With lime	Injury indicated	Spraying safe.
3	Without lime	Injured	Spraying unsafe.
	With lime	Injury indicated	Spraying safe.
4	Without lime	Badly injured	Spraying unsafe.
	With lime	Somewhat injured	Spraying questionable.
5	Without lime	Badly injured	Spraying unsafe.
	With lime	Injured	Do.
6	Without lime	Badly injured	Do.
	With lime	Injured	Do.

PEACH, VARIETY, STUMP.

No.	Mixture used.	Effect on foliage.	Remarks.
1	Without lime	Somewhat injured	Spraying questionable.
	With lime	Injury indicated	Spraying safe.
2	Without lime	Injured	Spraying unsafe.
	With lime	Injury indicated	Spraying safe.
3	Without lime	Injured	Spraying unsafe.
	With lime	Somewhat injured	Spraying questionable.
4	Without lime	Badly injured	Spraying unsafe.
	With lime	Injured	Do.
5	Without lime	Badly injured	Do.
	With lime	Injured	Do.
6	Without lime	Badly injured	Do.
	With lime	Injured	Do.

On June 16 and 17 another set of sprayings was made on the plum trees, the object being to obtain the cumulative effect of the sprays and also to ascertain whether the same injury would be done at this time as was noted after the sprayings of May 25 and 26. In this experiment the plum trees previously used were again sprayed with the same solutions and, in addition, some trees not used before were treated. Solutions Nos. 1, 2, and 3 were applied on the afternoon of June 16, and solutions Nos. 4, 5, and 6 on June 17. A rainfall of 0.19 inch occurred, however, on the same day, and the results with Nos. 4, 5, and 6 were entirely inconclusive, as no effect at all was produced, except on protected portions of the lower leaves.

The results obtained with Nos. 1, 2, and 3, applied on June 16 and followed by 0.19 inch of rain on the following day, while not definite enough for tabulation, plainly indicate that the last spraying did more injury than the first, and this was especially noted in the case of the trees which received two applications. The greater amount of injury resulting from the second spraying can be easily explained, as at the time of the first spraying the leaves were still young and the weather for four weeks previous had been extremely dry. Furthermore, from the weather chart it will be seen that only a small amount of rain fell during the three weeks following the spraying on May 25 and 26.

CONCLUSIONS.

The results obtained may be summarized as follows:

(1) Of the apple, pear, plum, and peach foliages the apple is injured the least, the pear and plum are affected to about the same degree, and the peach is injured the most.

(2) If only one application is made, Paris Green containing 4 per cent of free arsenious oxid can be applied with safety to apple trees and with the addition of lime 6 per cent may be present.

(3) If only one application is made to pear or plum trees, Paris Green containing 3 per cent of free arsenious oxid can be applied with safety and with the addition of lime 5 per cent.

(4) One application to peach trees of Paris Green containing 3 per cent of free arsenious oxid can not be made with safety without the addition of lime; with lime 4 per cent may be used.

(5) Two applications will do more damage than a single one, and more injury is apt to occur if the weather conditions were normal previous to the spraying than if very dry weather obtained.

Some observations were made on the manner in which the foliage of the different trees is affected. On the apple tree the injury is indicated by small, light-brown spots, well distributed over the leaves. On the pear and plum trees the damage is indicated by larger dark-brown spots, occurring mostly along the sides of the leaves, while on the peach leaves the spots are large, nearly circular, and distributed over various portions of the leaves.

REPORT FROM THE RHODE ISLAND STATION, KINGSTON.

By F. W. Card and A. E. Stene.

The spraying mixtures were made up strictly according to directions. The apple and pear trees were sprayed on May 25, 1903, and the peaches on May 27. The following table shows the weather conditions under which the experiment was conducted:

Meteorological data, Kingston, R. I.

Date.	Rainfall.	Character of day.	Date.	Rainfall.	Character of day.
1903.	*Inches.*		1903.	*Inches.*	
May 25	0.00	Clear.	June 5	0.00	Smoky.
26	.00	Do.	6	.00	Do.
27	.00	Partly cloudy.	7	.41	Cloudy.
28	.00	Cloudy.	8	.86	Do.
29	.00	Clear.	9	.00	Do.
30	.50	Partly cloudy.	10	.39	Do.
31	.00	Clear.	11	1.77	Do.
June 1	.00	Do.	12	.45	Do.
2	.00	Do.	13	.00	Clear.
3	.00	Do.	14	.00	Do.
4	.00	Smoky.			

OBSERVATIONS.

Apples.

The apple trees used for the experiment were Northern Spys, and one tree was sprayed with each sample of green. The trees stood in two rows, side by side, and the samples with lime were applied to one row and those without lime to the other. These experiments gave but slight results. Of the trees sprayed without lime the two receiving samples Nos. 2 and 5 (4 and 7 per cent of arsenious oxid) showed some burning of the foliage after the rains of June 7 to 10. The tree sprayed with No. 2 seemed to be the most affected. The trees sprayed with the addition of lime suffered no appreciable burning of the foliage.

Pears.

The pear trees receiving no lime were the Frederick Clapp, Hardy, Kieffer, Pitmaston Duchess, Superfine, and Anjou; those receiving the addition of lime were the Hardy, Hoosic, Louise Bonne, Sheldon, and

Urbaniste. These trees were all in a single row and every other one was sprayed with the samples containing no lime, the alternates being sprayed with the admixture of lime. After the rain of June 7 to 10 the Pitmaston Duchess, sprayed without the addition of lime, had a few burned leaves, and the Kieffer and Hardy, similarly treated, showed slight traces of burning. The trees receiving the spraying mixture mixed with lime showed no trace of burning whatever.

PEACHES.

The peaches sprayed stood in two rows, side by side, one row receiving the Paris Green alone and the corresponding trees in the other row receiving the same green with the addition of lime. Those receiving the samples with lime were three of the Crawford Late variety and three Fitzgeralds. Three Champions and three of the Hill Chili variety were sprayed with the samples without lime. None of the trees showed any effects of the spraying until after the rain of May 30. The results in every case were proportionate to the amount of soluble arsenious oxid used. Those receiving the lime were far less affected than those not receiving it; in fact, the former showed hardly any effects until after the heavy rains of June 7 to 10. The tree which was sprayed with sample No. 6 (8 per cent of arsenious oxid), without lime, lost nearly all of its leaves and did not recover during the season. A number of the branches died and it made practically no growth after the application of the green. The tree sprayed with the same sample with lime was affected about the same as the tree sprayed with sample No. 1 (2.84 per cent of arsenious oxid) without lime. As before stated, the other trees were affected proportionately to the amount of soluble arsenious oxid in the sample with which they were sprayed.

It appears that sample No. 3 (5 per cent of arsenious oxid) represents the maximum limit when lime is added, since Nos. 4, 5, and 6 (6, 7, and 8 per cent of arsenious oxid) caused considerable damage. All of the greens did too much injury to the peach trees when used without lime. In no case did the trees sprayed seem to lose their leaves any earlier than those not sprayed.

The illustrations accompanying this report show the injury to peach trees inflicted by several of the greens, and with their legends explain themselves.

REPORT FROM THE SOUTH CAROLINA STATION, CLEMSON COLLEGE.

By C. E. CHAMBLISS.

The solutions were made up and applied in strict conformity to the accompanying directions. The table following shows the condition of the weather during the time of the experiment.

Meteorological data, Clemson College, S. C.

Date.	Rainfall.	Maximum temperature.	Minimum temperature.
1903.	*Inches.*	°*F.*	°*F.*
June 12	00	75	58
13	00	74	42
14	00	80	47
15	00	89	54
16	00	89	54
17	00	88	56
18	00	91	58
19	00	91	60
20	00	94	62
21	00	....	....
June 22	T.	89	69
23	00	92	61
24	00	89	56
25	.08	90	60
26	.09	80	60
27	.10	80	66
28	00	85	62
29	00	87	66
30	00	93	62

OBSERVATIONS.

APPLES (WITHOUT LIME).

This group of trees was sprayed on June 15, 1903, and observations were made on June 23, up to which time no rain had fallen, and again on July 11, during which period there had been several rainfalls. At the first observation no effects were noticeable on any of the trees. At the second observation the following effects were noted:

No. 1. Simmons Red.—The edges of some leaves were black and many had fallen.

No. 2. Maverick Sweet and Laurence Greening.—Foliage of the former was black on the edges, but no leaves had fallen. About one-fifth of the foliage of the latter was black on the edges, but no leaves had fallen.

No. 3. Laurence Greening and Elgin Pippin.—The foliage of the former was badly burned and one-third of it had fallen; one-third of the foliage of the latter was burned very slightly.

No. 4. Mrs. Bryan and Shockley.—The foliage of the former was only slightly burned and a few leaves had fallen; the foliage of the latter was burned and the tree seemed to be sickly.

No. 5. Yopps Favorite and Golden Beauty.—One-half of the foliage of both had fallen and the rest was badly burned.

No. 6. Horn.—One tree suffered a slight burning of the foliage, while the other was practically unaffected.

APPLES (WITH LIME).

This group of trees was sprayed on June 16, 1903, and observations were made on the dates mentioned above. At the first observation no effect was noticeable, but at the second the following changes were noted:

No. 1. Black Warrior.—One-half of the foliage had fallen and the remainder was slightly burned.

No. 2. Rawls Janet.—Only a few leaves had fallen, the remainder being slightly burned.

No. 3. Grimes Golden.—These two trees were apparently not affected.

No. 4. Family and Rhodes Orange.—A few leaves had fallen.

No. 5. Red June.—One-third of the foliage had fallen and the remainder was burned.

No. 6. Jewetts Best and Chattahoochee.—One-fifth of the foliage of the former had fallen and the remainder was burned. The foliage of the latter was only slightly burned.

PEACHES (WITHOUT LIME).

These trees were sprayed and observations taken on the dates mentioned above for apples without lime.

No. 1. Tinley's October.—At the first observation a few leaves had fallen and at the second four-fifths of the foliage had fallen and the remaining leaves were badly burned.

No. 2. Tinley's October.—At the first observation a few leaves had fallen and at the second four-fifths of the foliage had fallen and the remaining leaves were badly burned, but new leaves were putting forth.

PEACHES (WITH LIME).

These trees were sprayed on June 16, 1903, and observations were taken on the dates mentioned above.

No. 1. Elberta and Stump the World.—At the first observation a few leaves had fallen from both. At the second observation the former had lost one-half of its foliage and the remainder was badly burned, while the latter had lost four-fifths of its foliage, the remainder being the terminals.

No. 2. Tinley's October.—At the first observation a few leaves had fallen. At the second observation four-fifths of the foliage had fallen, but new leaves were putting forth.

The stronger spraying solutions, Nos. 3 to 6, were not tested on the peach trees.

PLUMS (WITHOUT LIME).

The trees were sprayed on June 15, and observations taken on the dates mentioned above.

No. 3. Lombard.—At the first observation a few leaves were slightly burned. At the second observation one-third of the foliage had fallen. The leaves were badly burned on one tree and very yellow on the other. New leaves were putting forth.

No. 4. Chalco.—At the first observation the leaves fell readily when the tree was shaken. At the second observation only a few leaves remained on the trees.

No. 5. Ogon and Wanamaker. At the first observation yellow leaves were numerous and fell readily when the trees were shaken. At the second observation all of the leaves had fallen.

No. 6. German Prune and Juicy.—At the first observation no effect was noticeable. At the second both were badly burned, but practically no leaves had fallen.

PLUMS (WITHOUT LIME).

The trees were sprayed on June 16, 1903, and observations taken on the dates mentioned above.

No. 3. Shiro and Fellenburg.—At the first observation no effect was noticeable. At the second some of the foliage of the former had fallen and yellow and badly burned leaves were present. Only a few leaves of the latter tree had fallen, but they were badly burned.

No. 4. Red June.—At the first observation leaves fell when the tree was shaken. At the second nine-tenths of the foliage had fallen and the remaining leaves were badly burned.

No. 5. Wickson and Abundance.—At the first observation the leaves of the former tree fell readily when shaken, and burned leaves were numerous on the latter. At the second observation one-half of the foliage of both had fallen, but new leaves were putting forth.

No. 6. Wickson and Sultan.—At the first observation leaves fell when the trees were shaken. At the second one-third of the foliage had fallen, but new leaves were putting forth.

RESULTS.

These results, obtained as they were by one spraying of one or two trees by each sample, are not considered conclusive; a tabulation of the results of several sprayings would be more significant. However, from the results obtained it would appear that it is not safe to spray apple trees with Paris Green containing more than 4 per cent of soluble arsenious oxid without lime; with lime not more than 6 per cent should be present. None of the samples, either with or without lime, appears to be safe on peach foliage. The smallest percentage used on plum trees was 5 per cent of soluble arsenious oxid, and that was too strong without lime, but from the results obtained with 5 per cent of soluble arsenious oxid with lime, it would seem that 4 per cent with lime would be safe.

REPORT FROM THE NEW HAMPSHIRE STATION, DURHAM.

By C. M. Weed and J. L. Randall.

Plum and apple trees were used for this experiment, as they were the only ones available for the purpose at the time. All the trees were of thrifty growth, the apples of five years bearing and the plums of two years. They were all of the same age and practically the same height. The trees were sprayed on July 28, which was warm and clear, and the effect on the foliage, whenever there was any, was noted within less than twenty-four hours. The following notes were taken:

OBSERVATIONS.

Apples (without lime).

Baldwin apples were used exclusively both for the experiments with and without lime.

No. 1 showed several burnt spots, but was not severely injured and no leaves fell.

No. 2 did not differ from No. 1 and the spots were not very abundant.

No. 3, although there seemed to be no cause for it, was burned more than any other tree except No. 6. Brown blotches appeared on a large part of the foliage and a few leaves curled as if about to fall.

Nos. 4 and 5 both had many burned spots and some blotches. None of the leaves curled, except occasionally when a blotch or spot came to the edge of the leaf.

No. 6 was injured the most of any. Large patches appeared on many of the leaves, which turned brown. Under the microscope the cell protoplasm was shrunken in these blotches and in the centers of the smaller spots.

APPLES (WITH LIME).

No. 1 had small brown spots on a very few leaves.

No. 2 was burned more than No. 1, yet the leaves did not fall and the cells were killed only in a few places.

No. 3 suffered little, if any, more injury than No. 2.

Nos. 4 and 5 showed some burned spots on the leaves unevenly distributed. These trees were affected about the same as Nos. 1 and 2 without lime.

No. 6 showed smaller but more numerous spots than those observed on Nos. 4 and 5.

PLUMS (WITHOUT LIME).

No. 1. Imperial Sage.—The leaves showed quite prominent brown spots, which were more numerous in the direct sunlight than on shaded parts of the tree.

No. 2. Bradshaw.—This tree was slightly more damaged than No. 1, but none of the leaves was injured enough to fall.

No. 3. Abundance.—This tree was damaged more than any other except No. 6. Many of the leaves were curled and a few fell. The tree had not entirely recovered when the leaves fell in the autumn.

No. 4. Washington.—Some of the leaves were burned and curled, but the injury done was not so great as in the case of No. 3. The tree had recovered before the leaves fell.

No. 5. Yellow Japan.—A few leaves curled and fell. This tree was burned more than No. 4 and about the same as No. 3.

No. 6. Wyant.—This tree was burned more than any other. Many of the leaves at the top were half burned and a large number fell.

PLUMS (WITH LIME).

No. 1. De Soto.—On July 30 this tree showed only a few scattered brown spots on the leaves. On August 15 it had entirely recovered.

No. 2. German Prune.—The injury was about the same as that inflicted on No. 1, without lime. A few brown spots were observed, which gradually disappeared, until on August 21 the tree had entirely recovered.

No. 3. Reine Claude.—About as much damage was done in this case as to No. 2, without lime, the lime seeming to nullify the effect of about 1 per cent of soluble arsenious oxid. On August 25 the tree had entirely recovered.

No. 4. Green Gage.—The leaves of this tree were burned in round spots, and a few curled; but in thirty days it had entirely recovered.

No. 5. Satsuma.—This tree was damaged about the same as No. 4, without lime.

No. 6. Weaver.—Spots and blotches appeared, and a few leaves fell; but the tree recovered before the season was over.

The authors of the report from the New Hampshire station prefer not to give an opinion in regard to the amount of soluble arsenious oxid that can be endured by apple and plum trees, as the data obtained is deemed by them insufficient to serve as a basis of such an opinion. It appears, however, to the author that certain tentative conclusions may be drawn which can be compared with the results obtained by the various other stations and definite conclusions thus be obtained. Basing, then, an opinion upon this single set of experiments, it would appear:

(1) That when lime is not used a Paris Green containing 4 per cent of soluble arsenious oxid can be used on apple trees, while one containing 7 per cent can be used with the addition of lime.

(2) That if lime is not used a Paris Green containing 4 per cent of soluble arsenious oxid can be used on plum trees. If lime is used it would appear that a Paris Green containing 5 or 6 per cent might be safely used.

REPORT FROM THE OREGON STATION, CORVALLIS.

By C. B. Cordley.

In the experimental work done in testing the effects of varying amounts of free arsenious oxid in Paris Green on foliage, the directions were very carefully followed. For each test the required amount of water was placed in a barrel, to which was added the amount of milk of lime specified, if any, and then the Paris Green. The whole was stirred thoroughly and at once sprayed upon the trees. The work was done with a small hand pump, fitted with a Vermorel nozzle, and great care was exercised to see that every leaf was thoroughly sprayed. In addition to the work outlined the effect of the stronger sprays (1 ounce to 9.5 gallons) on the foliage of a few delicate plants was tested.

The sprays without the addition of lime were applied on July 10, 1903. Evaporation was so rapid that the foliage was dry a few moments after being sprayed. On July 11 the conditions were similar, and samples Nos. 1 to 3, with 15.5 gallons of water and 2 ounces of lime, were applied. The remainder of the samples of that set, Nos. 4 to 6, with 15.5 gallons of water and 2 ounces of lime, and Nos. 1 to 6, with 9.5 gallons of water and 2 ounces of lime, were applied on July 17.

In the meantime, i. e., from July 10 to July 17, the weather had remained clear and bright. A critical examination, made on the latter date, of the trees sprayed on July 10 and 11 showed that none of them was injured.

On July 21 and 23 there were slight showers, and cloudy weather continued throughout the week. Thorough examinations were made on July 23, August 3, and December 1, the results of which are given in the tabular statements which follow. A table is also given showing the meteorological conditions which obtained for a number of days following the application of the sprays.

Meteorological conditions at Corvallis, Oreg.

Date.	Temperature.		Precipitation.	Character of day.	Date.	Temperature.		Precipitation.	Character of day.
	Max.	Min.				Max.	Min.		
1903.	°*F.*	°*F.*	*Inch.*		1903.	°*F.*	°*F.*	*Inch.*	
July 10	86	48	0.00	Clear.	July 21	70	50	0.04	Cloudy.
11	70	51	.00	Do.	22	72	47	.00	Do.
12	74	47	.00	Do.	23	66	52	.05	Do.
13	70	52	.00	Do.	24	74	59	.00	Do.
14	77	47	.00	Do.	25	75	47	.00	Partly cloudy.
15	86	48	.00	Do.	26	77	42	.00	Do.
16	81	50	.00	Do.	27	83	45	.00	Clear.
17	82	47	.00	Do.	28	84	45	.00	Do.
18	75	48	.00	Do.	29	77	54	.00	Partly cloudy.
19	81	50	.00	Do.	30	75	55	.00	Clear.
20	86	50	.00	Do.	31	77	46	.00	Do.

OBSERVATIONS.

TABLE I.—APPLES WITHOUT LIME.

[1 ounce to 9.5 gallons.]

Sample No.	Variety.	Injury.		
		July 23.	August 3.	December 1.
1	Mackintosh	None	None	But few leaves fallen.
	Carolina June	.....do	.....do	Do.
	Tuft's Baldwin	.....do	.....do	Do.
2	Benoni	.....do	.....do	Do.
	Crab	.....do	.....do	Completely defoliated.
	Spitzenberg	.....do	.....do	No leaves fallen.
3	.....do	.....do	.....do	Few leaves fallen.
	.....do	.....do	.....do	Do.
	Spokane Beauty	.....do	.....do	Do.
4	.....do	.....do	.....do	Do.
	.....do	Few leaves spotted	No increase of spots	Nearly defoliated.
5		None	None	Completely defoliated.
	Yellow Transparent	.....do	.....do	Half of leaves fallen.
	Grimes' Golden	.....do	.....do	Completely defoliated.
6	Hyde's King of West	Few leaves spotted	No increase of spots	Almost defoliated.
	Langford	Few leaves spotted and few killed.	No increase of injury.	Half of leaves fallen.
	Yakama	.....do	.....do	Few leaves fallen.
	Yellow Newton Pippin.	.....do	.....do	Completely defoliated.

Observations—Continued.

APPLES, WITH LIME.

Sample No.	Variety.	Injury. July 23.	Injury. August 3.	Injury. December 1.
1	Rome Beauty	None	None	But few leaves fallen.
	....do	....do	....do	Do.
	....do	....do	....do	Half of leaves fallen.
2	....do	....do	....do	Completely defoliated.
	Waxen	....do	....do	But few leaves fallen.
	....do	....do	....do	Nine-tenths of leaves fallen.
3	....do	....do	....do	Do.
	....do	....do	....do	Do.
4	....do	....do	....do	Three-fourths of leaves fallen.
	....do	....do	....do	Do.
	....do	....do	....do	Seven-eighths of leaves fallen.
5	....do	....do	....do	Few leaves fallen.
	....do	....do	....do	Three-fourths of leaves fallen.
6	....do	....do	....do	Seven-eighths of leaves fallen.
	....do	Few leaves slightly spotted, and a few more killed.	No increase of injury.	Do.

TABLE II.—PEARS, WITHOUT LIME.

[1 ounce to 9.5 gallons.]

1	Directeur Alphando	None	None	Completely defoliated.
	Colonel Wilder	....do	....do	But few leaves fallen.
2		....do	....do	Completely defoliated.
3	Bartlett	....do	....do	Few leaves fallen.
4		....do	....do	Do.
	Patrick Barry	....do	....do	Do.
5	Winter Bartlett Fame	Leaves slightly injured about margins of holes eaten by slugs.	No increase of injury.	Completely defoliated.
6	Genesta	None	None	Do.

PEARS, WITH LIME.

1	Cockling	None	None	Tree dead from other causes.
3	Buffum	....do	....do	Completely defoliated.
	....do	....do	....do	Do.
4	Lincoln	....do	....do	But few leaves fallen.
5	Genesta	....do	....do	Do.
6	....do	....do	....do	Seven-eighths of leaves fallen.
	Assumption	....do	....do	Completely defoliated.

Observations—Continued.

TABLE III.—PLUMS,[a] WITHOUT LIME.[b]

[1 ounce to 9.5 gallons.]

Sample No.	Variety.	Injury.		
		July 23.	August 3.	December 1.
1		None	None	Completely defoliated.
3		....do	....do	Do.
		....do	....do	Do.
		....do	....do	Do.
4		....do	....do	Do.
		....do	....do	Do.
5		....do	....do	Do.
6	Herefordshire	....do	....do	No record.

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[a] These trees were all prune-bearing plum trees, and were reported under the former designation; but the term "plum" is here used to bring them into comparison with the other plum trees used in the experiment.

[b] For the report on plums sprayed with the addition of lime see text at close of tables, page 29.

TABLE IV.—PEACHES, WITHOUT LIME.

[1 ounce to 15.5 gallons.]

Sample No.	Variety.	Injury.		
		July 23.	August 3.	December 1.
1	Old Mixon	None	None	Three-fourths of leaves fallen.
2	....do	Slightly injured	No increase of injury.	One-fourth of leaves fallen.
3	....do	....do	....do	One-half of leaves fallen.
4	....do	Considerably spotted.	One-fourth of leaves fallen; the rest injured.	Do.
5	Mont Rose	....do	....do	Do.
6	....do	Badly injured	One-half of leaves fallen; the rest injured.	
	....do	....do	....do	Do.

PEACHES, WITH LIME.

Sample No.	Variety.	July 23.	August 3.	December 1.
1	Lewis's Seedling	None	None	Three-fourths of leaves fallen.
	Imperial	Badly injured	One-half of leaves fallen.	One-fourth of leaves fallen.
2	....do	Slightly injured	No increase of injury.	Do.
	....do	....do	....do	Do.
3	Mumé	Very slightly injured.	....do	Do.
	....do	....do	....do	Do.
4	Alexander	None	None	Seven-eighths of leaves fallen.
		....do	....do	Three-fourths of leaves fallen.
5		....do	....do	Do.
	Willett	....do	....do	Do.
6	Old Mixon	....do	....do	No record.

Observations—Continued.

TABLE V.—PLUMS, WITHOUT LIME.

[1 ounce to 15.5 gallons.]

Sample No.	Variety.	Injury.		
		July 23.	August 3.	December 1.
1	Imperial Epineuse	None	None	Almost entirely defoliated.
	....do	....do	....do	Do.
2	....do	....do	....do	Do.
	....do	....do	....do	Do.
3	....do	....do	....do	Do.
	Willamette	....do	....do	Do.
4	....do	....do	....do	Do.
	....do	....do	....do	Completely defoliated.
5	....do	....do	....do	Almost entirely defoliated.
	....do	....do	....do	Completely defoliated.
6	....do	....do	....do	Do.
	Robe de Sargent	....do	....do	Do.

PLUMS (WITH LIME).

Only three trees were sprayed with the solutions made up with 9.5 gallons of water, Nos. 2, 3,[a] and 4 being used. No effects from the sprays were to be observed on either July 23 or August 3. On December 1 the first two were entirely defoliated, while the tree treated with No. 4 had only lost a few leaves.

A number of varieties were sprayed with the solutions Nos. 1 to 6, made up with 15.5 gallons of water to an ounce of the green. None of the trees showed any injury on either July 23 or August 3, but on December 1 all were either completely, or almost completely, defoliated. A Reine Claude plum tree sprayed with No. 1 and a Green Gage sprayed with No. 3 were among this group, with results as stated.

DISCUSSION.

The observations made on December 1 were for the purpose of finding out whether the sprayed trees dropped their leaves earlier in the fall than those not sprayed, and whether this premature falling, if it existed, had any relation to the amount of soluble arsenious oxid used on the tree. The time at which the leaves fall ordinarily varies greatly with the season and the variety of the tree. The college orchard consists of two or three trees each of quite a number of varieties, and when they were examined, on December 1, there was not only a surprising degree of variation in the condition of the foliage of the different varieties but also a great difference between the several trees of the same variety. Two apple trees (Waxen) standing side by side were sprayed with No. 2 plus lime. On December 1 only a few leaves had fallen from one of the trees while on the other not over 10 per cent remained. Likewise two trees of the same variety, standing side by side in the same row, were sprayed with No. 5 plus lime. When examined three-fourths of the leaves had fallen from one and only a few from the other. Thus the examination of the orchard failed to show that the spray had any effect whatever upon the falling of the foliage. The tabulated data, however, seem to indicate that a somewhat larger proportion of leaves had fallen by December 1 from trees sprayed with Nos. 5 and 6 than from those treated with Nos. 1, 2, 3, and 4. Whether this was due to the sprays or to mere chance in the selection of the trees, it is impossible to say. Little effect if any was produced on the apple, pear, and plum trees.

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[a] A cherry tree sprayed with No. 3 without the addition of lime showed no injury on July 23 and August 3, and on December 1 it was entirely defoliated.

As to the effects of the various strengths of soluble arsenious oxid the results of this set of experiments alone seem to show that under favorable conditions, such as prevailed at the time the spraying was done and during the following 30 days, it is safe to spray apple, pear, and plum trees with any of the 6 samples, both with and without the addition of lime. None of the samples injured any of the varieties of these fruits sufficiently for the damage to be considered of practical importance in orchard work. It may be noted, however, that the work was done under peculiarly favorable conditions. Probably not over 15 minutes elapsed in any case from the time the poison was first mixed in the water to the time it was sprayed on the trees, giving but a short time for the arsenious oxid to enter into solution, and the weather conditions were such that after being sprayed on the trees the moisture evaporated very rapidly. Under practical orchard conditions it would probably be safer not to use Nos. 5 and 6 on apple, plum, or pear trees without the addition of lime.

No conclusions can be drawn from this series of experiments as to the peach trees, as these trees in the college orchard are growing on soil entirely unsuited to them. The trees are not thrifty, some of them are nearly dead, and it is believed that the resistance of the foliage of the various trees to the action of the soluble arsenic varies greatly.

SUMMARY.

The following table shows the maximum amount of soluble arsenious oxid in Paris Green which can be safely used on the varieties of fruit trees included in these experiments. It must be remembered when 8 per cent is given as the maximum that this was the strongest solution used, and had stronger ones been employed they might have been found safe also for such trees as endured the 8 per cent solution of soluble arsenious oxid, and these cases are therefore marked "8+." If even the smallest amount of soluble arsenious oxid was found to be injurious the word "inapplicable" is used, since the sample marked No. 1 is as pure a Paris Green as can be obtained on the market.

Amounts of soluble arsenious oxid allowable in Paris Green.

[Samples analyzed by ten-day water-extraction method.]

Place of experiment.	Apple.		Pear.		Peach.		Plum.	
	Without lime.	With lime.	Without lime.	With lime.	Without lime.	With lime.	Without lime.	With lime.
	Per cent.	*Per ct.*	*Per cent.*	*Per ct.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per ct.*
Washington, D. C	8+	8+	8+	8+	Inapplicable.	6	....	....
College Park, Md	8+	8+	8+	8+	....do...	5	4	5
Ithaca, N. Y	4	6	3	5	....do...	4	3	5
Kingston, R. I	6	8+	8+	8+	....do...	5	....	....
Durham, N. H	4	7	....	....	....	....	4	5-6
Clemson College, S. C	4	6	....	....	Inapplicable.	Inapplicable.	....	4
Corvallis, Oreg	8+	8+	8+	8+	....	....	8+	8+

A study of the weather charts for the various stations shows that the following periods elapsed between the dates of spraying and the first rainfall: Washington, D. C., about 10 days; College Park, Md., about 12 days; Ithaca, N. Y., about 3 days before a very light rain and then 13 days before the next rainfall; Kingston, R. I., about 5 days and then 7 more before another rain; Clemson College, S. C., 10 days, followed by a very light rainfall and a long dry term. It is thus seen that in nearly all cases the weather conditions were almost ideal, if the word may so be used, for producing the maximum damage from the sprays. During the rather long dry period after spraying, when only light rains fell, if any, the dew and carbon dioxid of the air acted upon the Paris Greens, causing them to decompose and set free the arsenious oxid, so that when rain finally came a large amount of soluble arsenious oxid was present, which the leaves absorbed and thus caused burning. These conditions were especially striking at Clemson College, where no rain fell for 10 days, after which just about enough rain fell for 3 days to dissolve the soluble arsenious oxid and yet not wash it off. Another dry period followed, terminated by rains, which caused the soluble arsenious oxid to burn the foliage.

Taking the weather conditions into consideration, and judging the results in a very conservative manner, it would appear from the figures given in the table that the following conclusions may be drawn:

(1) For apple trees sprayed without the use of lime the amount of soluble arsenious oxid[a] allowable lies between 4 and 8+ per cent, a fair average being 6 per cent.

(2) For apple trees sprayed with the use of lime the amount of soluble arsenious oxid allowable is between 6 and 8+ per cent, a fair average being 7 per cent.

(3) For pear trees sprayed without the use of lime the amount of

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[a] By soluble arsenious oxid is meant the amount extracted from 1 part of Paris Green by 1,000 parts of carbon dioxid-free water during the course of 10 days.

soluble arsenious oxid allowable is between 3 and 8+ per cent, a fair average being 6 per cent.

(4) For pear trees sprayed with the use of lime the amount of soluble arsenious oxid allowable is between 5 and 8+ per cent, a fair average being 7 per cent.

(5) For plum trees sprayed without the use of lime the amount of soluble arsenious oxid allowable is between 3 and 8+ per cent, a fair average being about 4 per cent.

(6) For plum trees sprayed with the use of lime the amount of soluble arsenious oxid allowable is between 4 and 8+ per cent, a fair average being from 5 to 6 per cent.

(7) In spraying peach trees none of the Paris Greens bought upon our market should be used without the addition of lime.

(8) For peach trees sprayed with the use of lime the amount of soluble arsenious oxid allowable lies between 3 and 6 per cent, a fair average being 4.5 per cent.

(9) The difference in the effect produced by Paris Greens used with and without the addition of lime is so great that lime should always be employed.

For ready reference these conclusions are also embodied in the following table:

Maximum and average amounts of soluble arsenious oxid allowable.

Apple.				Pear.			
Without lime.		With lime.		Without lime.		With lime.	
Limits.	Average.	Limits.	Average.	Limits.	Average.	Limits.	Average.
Per cent. 4-8+	*Per cent.* 6	*Per cent.* 6-8+	*Per cent.* 7	*Per cent.* 3-8+	*Per cent.* 6	*Per cent.* 5-8+	*Per cent.* 7

Peach.				Plum.			
Without lime.		With lime.		Without lime.		With lime.	
Limits.	Average.	Limits.	Average.	Limits.	Average.	Limits.	Average.
Per cent. 0-0	*Per cent.* 0	*Per cent.* 3-6	*Per cent.* 4.5	*Per cent.* 3-8+	*Per cent.* 4	*Per cent.* 4-8+	*Per cent.* 6

It is evident that the amount of soluble arsenious oxid allowable in a Paris Green depends largely on the kind of fruit tree that is to be sprayed. The author is of the opinion that State chemists should not select any fixed standard by which to judge all Paris Greens sent in by the farmer, but should find out for what purpose the green is to be used and base his decision on the resisting power of the kind of tree to be sprayed. It would perhaps be advisable to have a printed form giving the names of the various common fruit trees and to state after each whether or not the sample sent could be used upon it.

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